HYDROGEN SULFIDE (H_2S) ADSORPTION BY POLYETHYLENE FOAM

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Abstract

In this present work, the removal of hydrogen sulfide (H_2S), which is generated from the biogas production, by the adsorption process with polyethylene foam as an adsorbent is studied. This study used Fe_2O_3-PE foam to adsorb hydrogen sulfide by compounded Fe_2O_3 with polyethylene (PE) and formed into the foam. Then the Fe_2O_3-PE foam have been tested the H_2S removal performance by packing the foam in the batch adsorption column to find the H_2S adsorption rate. The morphology of the Fe_2O_3-PE foam was analyzed by SEM analysis to investigate the morphology that suitable for the reaction between Fe_2O_3 and H_2S in the foam. The reaction of Fe_2O_3 and H_2S at the foam surface and inside the foam. The result shows that the 60 phr of Fe_2O_3 foam found to have the best H_2S adsorption performance due to the uniform dispersion of Fe_2O_3 in the PE and it also has thin cell wall (3-5 microns). The amount of foam used in the H_2S adsorption where bulk density 0.015 g/cm^3, 0.030 g/cm^3 and 0.015 g/cm^3 with the presence of water. The result also shows that the reaction between Fe_2O_3 and H_2S is second-order and have the reaction rate constant of 1.2×10^{-5} min^{-1}, 1.8×10^{-5} min^{-1} and 4.0×10^{-6} min^{-1} at room temperature.

Keywords: Fe_2O_3-PE foam, H_2S adsorption, H_2S removal

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บทคัดย่อ

งานวิจัยนี้ศึกษาการดูดซับแก๊สไฮโดรเจนซัลไฟด์ (H_2S) ที่เกิดขึ้นจากกระบวนการผลิตก๊าซชีวภาพ (biogas) และในบ่อบำบัดน้ำเสียของโรงไฟฟ้าก๊าซชีวภาพ โดยศึกษาการดูดซับแก๊สไฮโดรเจนซัลไฟด์ด้วยโฟม Fe_2O_3-PE จากการนำเหล็กออกไซด์ (Fe_2O_3) และโพลีเอทิลีน (PE) มาผสมและขึ้นรูปเป็นโฟม คอมปาวด์ระหว่าง Fe_2O_3-PE ในสัดส่วนที่ต่างกันได้ถูกศึกษาเพื่อดูสัดส่วนที่เหมาะสมในการขึ้นรูปเป็นโฟม จากนั้นโฟม Fe_2O_3-PE จะถูกนำไปทดสอบประสิทธิภาพในการกำจัดแก๊สไฮโดรเจนซัลไฟด์ โดยนำไปโฟมแพคและทดสอบการดูดซับแก๊สในคอลัมน์แบบกะ เพื่อหาอัตราการดูดซับแก๊สไฮโดรเจนซัลไฟด์ ลักษณะโครงสร้างของโฟมจะถูกวิเคราะห์โดย SEM เพื่อดูลักษณะโครงสร้างที่เหมาะสมสำหรับการทำปฏิกิริยาระหว่าง Fe_2O_3 และ H_2S ภายในโฟม รวมถึงวิเคราะห์การเกิดปฏิกิริยาระหว่าง Fe_2O_3 และ H_2S บริเวณผิวโฟมและด้านในโฟม และบริเวณที่มีการถ่ายเทมวล (mass transfer zone, MTZ) ผลจากการศึกษาพบว่าคอมปาวด์ที่มีสัดส่วน 40, 50 และ 60 phr of Fe_2O_3 สามารถคอมปาวด์และขึ้นรูปเป็นโฟมได้ ทั้งนี้พบว่าโฟมที่มีสัดส่วน 60 phr Fe_2O_3 สามารถดูดซับแก๊สไฮโดรเจนซัลไฟด์ได้มากที่สุดเนื่องจากมีการกระจายตัวของ Fe_2O_3 ในเนื้อ PE ที่สม่ำเสมอ และมีผนังเซลล์บางที่สุด (3-5 ไมครอน) โดยโฟมจะถูกนำมาทดสอบการดักจับ H_2S ที่ปริมาณอัตราความหนาแน่นการรวม(bulk density) 0.015 g/cm^3, 0.030 g/cm^3 และ 0.015 g/cm^3 ในสภาวะที่มีน้ำ พบว่าเป็นปฏิกิริยาอันดับสองและมีค่าคงที่ของอัตราการเกิดปฏิกิริยาเท่ากับ $1.2 \times 10^{-5} min^{-1}$, $1.8 \times 10^{-5} min^{-1}$ และ $4.0 \times 10^{-6} min^{-1}$

คำสำคัญ: Fe_2O_3-PE โฟม, การดูดซับก๊าซไฮโดรเจนซัลไฟด์, การกำจัดก๊าซไฮโดรเจนซัลไฟด์

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NOMENCLATURE

ΔH	=	Heat of reaction
ΔG	=	Gibbs free energy
k	=	Reaction rate constant
C	=	Concentration at time
C_0	=	Initial concentration
K_c	=	Equilibrium constant
D	=	Diffusivity
D_0	=	Pre-exponential factor
E_D	=	Activated energy of diffusion
R_g	=	Universal gas constant
T_g	=	Glass transition temperature
b	=	The thickness of cell wall/2
V_F	=	Polymeric foam volume
V_P	=	Polymer volume
ρ_F	=	Density of foam
ρ_P	=	Density of polymer
$\emptyset_P$	=	Expansion ratio
σ_P	=	Potential length constant
ε_P	=	Potential energy contant
PE	=	Polyethylene

INTRODUCTION

1.1 Background

Thailand is one of the countries that concerns about industrial waste management. The waste treatment process is one of the significant processes for all industries, however, some treatment process has high difficulty and require many steps. These problems could lead to an increase in operating cost and complication of the process. Therefore, the decrease in waste disposal cost could make the process more cost-effective and promote economic growth according to the higher investment in the country. In addition, the effective waste management process could minimize the effects to human and environment.

Wastewater from industries contain different chemical compositions depending on the type of industry [1] such as metal industry, painting industry, petroleum industry. The industry that contains livestock feed, agricultural solid waste or harvesting, often generate biogas via biogas fermentation system. Biogas is one of the renewable energies that can be produced by decomposing of non-oxygenated materials. The conventional biogas production method is gas storage. Biogas is widely used in power plants to produce electric power. The main components of biogas are methane; 50-70% and other components such as carbon dioxide, nitrogen, hydrogen, oxygen, and hydrogen sulfide.

The main problem of electric power production from biogas is the presenting of hydrogen sulfide (H_2S) because of its high toxicity and it can cause many health effects. H_2S content in the process should be less than 4 parts per million (ppm) [2]. The conventional process [3] for removing H_2S from wastewater is the adsorption process, which has been widely used due to the high performance on removing H_2S and cost saving. Ferric oxide (Fe_2O_3) can be used to remove H_2S by converting it to ferric sulfide (FeS). The reaction occurs when ferric sulfide (FeS) react with oxygen or air and convert to ferric oxide (Fe_2O_3). Nowadays, there are various types of commercial Fe_2O_3 adsorbent such as Media-G2, CG-4 and Activated Carbon[4], which made of porous medium with high porosity. These adsorbents usually packed in the adsorption column.

Some polymer foam such as polyethylene foam is considered as a good adsorbent for Fe_2O_3, since its pores has high acid/base stability and made cost saving. The durability of the polymer foam is ideal for trapping H_2S because it floats in high temperature and acidic wastewater. In this study, polyethylene foam (PE) and internal oxide is applied for the absorption process of H_2S substance from industrial effluent.

The reduction of hydrogen sulfide gas in the industrial effluent by using polyethylene foam with internal oxide is the expected result. Most of polyethylene beads and ferric oxide powder mixture were perfectly compounded at the mixing ratio of 40, 50 and 60 phr of Fe_2O_3. Afterward, it is transformed into Fe_2O_3-PE foam by adding carbon dioxide during the foaming process. The physical properties and H_2S removal efficiency are monitored by H_2S gas detector. Fe_2O_3-PE foam analysis is performed by scanning electron microscope (SEM).

According to the advantages of using PE-foam to adsorb H_2S, this research was conducted to investigate the factors that affects to the H_2S trapping performance. The optimal amount of Fe_2O_3 in the Fe_2O_3-PE foam, considered from the efficiency of foam formation and H_2S trapping efficiency, is investigated in this study. Moreover, the favorable condition for H_2S trapping is also investigated. The knowledge that has been used for the design and prototype of laboratory products is also applied to develop the laboratory-scale product into ready-to-use products for commercial use in the industrial level.

1.2 Objective(s)

1.2.1 To develop the knowledge on H_2S absorption process using PE-foam and advanced this process to be highly-effective H_2S trapping method.
1.2.2 To study polymeric foam processing and equipment.
1.2.3 To apply this study to industrial scale.

1.3 Scope(s) of Work

1.3.1 Find the suitable mixing ratio of polyethylene beads and ferric oxide powder mixture for polymer foaming process.
1.3.2 Apply the laboratory-scale process with high H_2S content removal to the industrial-scale process.

1.4 Expected Outputs

1.4.1 High pore volume and uniform of pore size of Fe_2O_3-PE foam.
1.4.2 Considerable adsorption performances.

CHAPTER II
LITERATURE REVIEW

2.1 Introduction of hydrogen sulfide

Hydrogen sulfide (H_2S) could be released from natural and industrial sources, distillation process and coal gasification process. It affects and damages the pipeline and the catalyst. To prevent the process and environmental impact, the minimum H_2S concentration should be less than 4 ppm[2] because it can react with rain and could convert to acid rain. Sulfur removal process has been developed and implemented in various ways. The appropriate H_2S removal methods are adsorption, absorption and biological method. The most widely used method is adsorption because they are highly effective and viral for use.

There are many ways to get rid of H_2S. The priority factors must be considered in order to keep the amount of H_2S at an acceptable level and still in legal. In order to eliminate H_2S, the cost of disposal must be taken into account, including the impact on the environment. Each type of H_2S removal process has different advantages and disadvantages. In the biogas industry and other industries with H_2S emissions need to have H_2S removal process. The amount of H_2S must be at the legal level before being released into public water.

The effective H_2S removal process will be used and further developed. These processes must be able to assess the cleaning efficiency in numerical terms. The criteria will be used to evaluate those performances. It is based on criteria such as the general value of the process. In order to choose the process to be used in wastewater treatment, it is necessary to analyze the properties of the process. In order to get the most efficient wastewater treatment process. The H_2S removal process is as follows.

2.1.1 Hydrogen sulfide properties

Characteristics of H_2S are colorless, extremely toxic and flammable. Identification bad odor same as the rotten eggs. The effect of the H_2S is a headache, cough, eye irritation, and insomnia by exposure small doses. Human will be fainting in five minutes by 1,000 ppm exposure[5]. H_2S gas can slightly be dissolved in water and its solution is highly corrosive (pH value around 4.5) to entire equipment, production pipelines and toxic to the catalysts. General physic properties of H_2S is shown in Table 2.1.

Table 2.1 Hydrogen sulfide properties

Hydrogen sulfide properties	
Formula	H_2S
Molecular Weight	34.08
Boiling Point	-60.4 °C
Density/Specific Gravity	1.539 g/L at 0 °C (water = 1)
Vapor Pressure	20 atm at 25.5 °C

2.1.2 Hydrogen sulfide content in bio gas

The fermentation process mainly produces methane gas of biogas generation. The biogas is contaminated by the small content of H_2S concentration approximately 10-500 ppm[6].

2.2 Hydrogen sulfide removal process

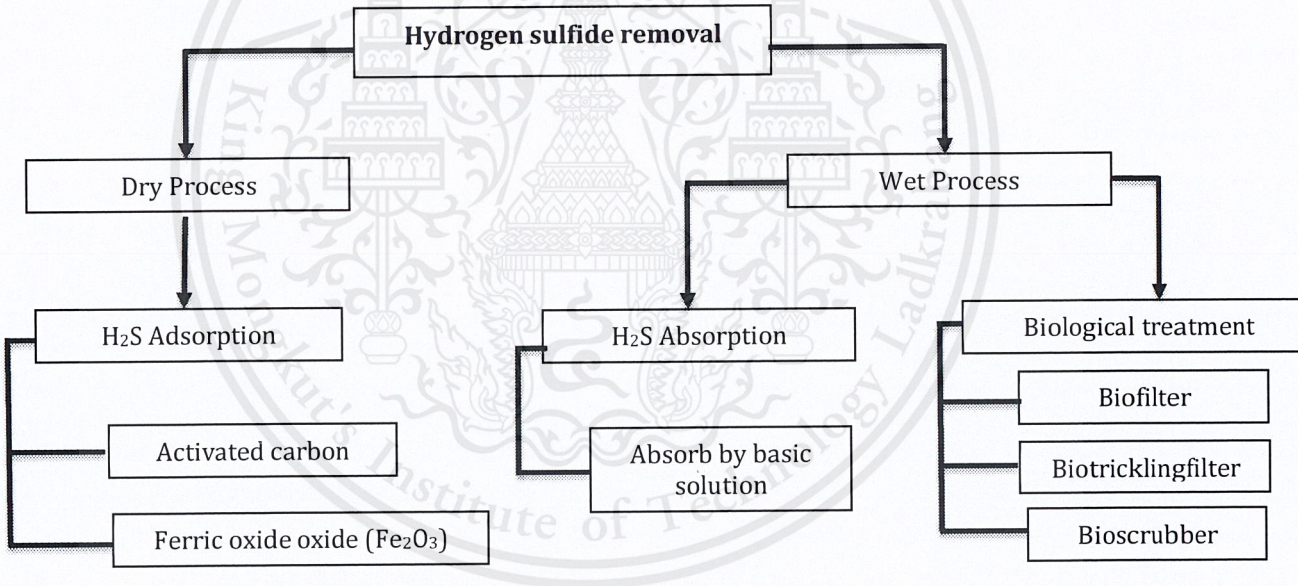


Figure 2.1 Diagram of hydrogen sulfide removal process

The hydrogen sulfide removal process can be divided into two types which are dry process and wet process. The dry process could adsorb H_2S by using activated carbon, ferric oxide (Fe_2O_3), etc, which would be explained in the next step. The wet process is separated into two types which are H_2S absorption and biological treatment. The H_2S absorption use basic solution to absorb H_2S, which would be explained further in the next topic. The biological treatment can remove around 99%[7] of H_2S by bio filter, biotricklingfilter and bioscrubber.

2.2.1 Dry process

The dry process is a process of adsorbing hydrogen sulfide by using adsorbents such as activated carbon and Ferric oxide (Fe_2O_3). The contaminated substance with hydrogen sulfide gas is adsorbed by the adsorbent. After the adsorbent completely adsorbs the hydrogen sulfide gas, the adsorbent will be regenerated by air.

1. Hydrogen sulfide adsorption process

Chemical removal process by H_2S adsorption generally is accomplished by using metal oxides such as ferric oxide, zinc oxide, barium oxide, zirconium oxide and etc[8][2]. These metal oxides reactions were studied wildly disseminate. Several equations of reaction are listed below.

$$Fe_2O_3\ (s) + 3H_2S\ (g) \rightarrow Fe_2S_3\ (s) + 3H_2O\ (l) \quad (2.1)$$

$$CaO\ (s) + H_2S\ (g) \rightarrow CaS\ (s) + H_2O\ (l) \quad (2.2)$$

$$ZnO\ (s) + H_2S\ (g) \rightarrow ZnS\ (s) + H_2O\ (l) \quad (2.3)$$

$$BaO\ (s) + H_2S\ (g) \rightarrow BaS\ (s) + H_2O\ (l) \quad (2.4)$$

Hydrogen sulfide adsorption processes are widely used because of simple operation. That occurs by contracting between adsorbents and hydrogen sulfide constituent. Air with hydrogen sulfide constituent was passed to adsorbents column. Adsorbent selection is important to evaluate eventually saturated of adsorbent media. Adsorbent column generally consists of two vessels or more than; one of them is on the process. While other is offline for regeneration. Absorption processes can be classified for physical absorption and chemical absorption.

However, the conditions of the process should be considered; ZnO reactions is proper for operation temperature lower than 100°C and for CaO reactions is approximately 250-500°C. Ferric oxide (Fe_2O_3) is the best choice of the metal oxide for H_2S removal at room temperature operation that is going to describe in next topic.

H_2S adsorption process need to concentrate physical properties such as high surface area and high pore volume are desired. Adsorbent property for physical adsorption is important molecular sieves (zeolites) which high surface area and pore size. It can be generated in natural or synthesized by silicates/aluminate.

2.2.2 Wet process

Wet process is a process of absorbing hydrogen sulfide by using stripping process and biological treatment. Stripping process of H_2S remove sour gas by stripping H_2S into the aqueous solution. Afterward, the suitable process to remove H_2S needs to be considered. One method is biological treatment using bacteria, which results in 99-100% of H_2S removal.

1. Stripping process

This process uses an aqueous solution to remove hydrogen sulfide. In this system, hydrogen sulfide gas flows up through the column. Hydrogen sulfide is captured by an aqueous solution. The aqueous solution after process needs to be regenerated by air.

Stripping process in industrial use water, MEA, and DEA in the process because of the commercial prices and easy source. Although H_2S is slightly dissolved into water (0.4 %wt/wt at 20°C) but it is still disposal in process. MEA and DEA are used in industrial better than use water. The equation of hydrogen sulfide removal process is:

$$H_2S\ (g) \rightarrow H_2S\ (aq) \leftrightarrow H^+ + HS^- \qquad (2.5)$$

H_2S in an aqueous phase change to H^+ and HS^-. The efficiency absorption will increase by reducing of these two mentioned ions.

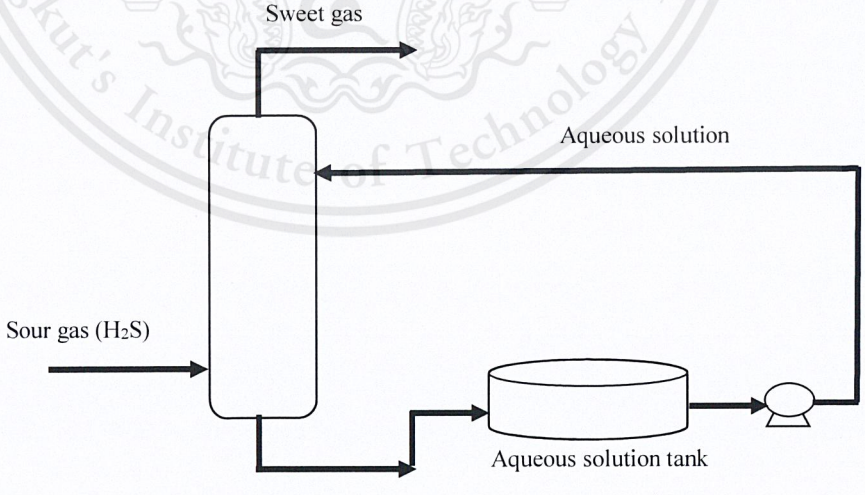


Figure 2.2 Block flow diagram of stripping process

2. Biological treatment

In the biological treatment, H_2S is removed by some species of bacteria via oxidation reaction. These bacteria are coated as film cover bio-filter. The aerobic conditions absorb H_2S each layer. Hydrogen sulfide is removed around 99% was obtained by using this method. This solution is highly-effective but high operational cost.

$$H_2S\,(g) + 2O_2\,(g) \xrightarrow{\text{bacteria}} H_2SO_4\,(aq) \qquad (2.6)$$

Another biological treatment method occurs in an anaerobic system with the light requirement by the reaction below[9]. This method founds to remove H_2S in the system nearly 100%.

$$2H_2S\,(g) + CO_2\,(g) \xrightarrow[\text{bacteria}]{\text{light}} 2S + (CHO) + H_2O\,(l) \qquad (2.7)$$

2.3 Introduction of ferric oxide

Ferric oxide or iron (III) oxide is an inorganic compound. It is one of the oxides of iron. They have three main types. First, ferric (III) oxide (Fe_2O_3) is generally known as "rust" or hematite mineral. Second, ferric (II) oxide (FeO) is rarely found in nature. Last is ferric (II, III) oxide (Fe_2O_3) found in magnetite mineral [10].

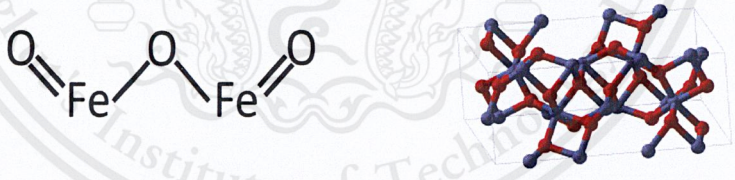


Figure 2.3 Ferric oxide structure

Table 2.2 Ferric oxide properties

Ferric oxide Properties	
Formula	Fe_2O_3
Molecular Weight	159.69
Appearance	Reddish-brown powder
Melting Point	1566 °C
Solubility in H_2O	Insoluble
Specific Heat	103.9 J/mol·K

Ferric oxide or iron (III) oxide for iron steel production by this equation.

$$Fe_2O_3\,(s) + 3CO\,(g) \rightarrow 2Fe\,(s) + 3CO_2\,(g) \quad (2.8)$$

Fe_2O_3 can be distinguished into various polymorph which dominates types are α-Fe_2O_3 and γ-Fe_2O_3. The α-Fe_2O_3 type is easier found in nature than γ-Fe_2O_3 because of γ-Fe_2O_3 must be covered α-Fe_2O_3 at high temperature or deriving from magnetite mineral.

Figure 2.4 (a) Alpha phase ferric (III) oxide[10] and
(b) gramma phase ferric (III) oxide[11]

Fe_2O_3 can also be prepared by this chemical reactions.

$$4Fe\,(s) + 3O_2\,(g) + 2H_2O\,(l) \rightarrow 4Fe(O)OH\,(s) \quad (2.9)$$

$$2Fe(O)OH\,(s) \rightarrow Fe_2O_3\,(s) + H_2O\,(l) \quad (2.10)$$

They have different colors because ferric oxide can form in many ways. For example, typical Fe_2O_3 is red iron which can turn to black as Fe_2O_3 at high temperature or iron hydroxide ($Fe(OH)_3$) in nature has yellow iron.

2.3.1 Hydrogen sulfide removal by ferric (III) oxide

Sulfur removal by ferric (III) oxide (Fe_2O_3) has been developed since the 19th Century[9]. The reaction is stable at the condition of room temperature that was acceptable for use in many industries. Sulfur in the gas stream will react with Fe_2O_3. Sulfur removal can observe by color changing of from either red or yellow to black color of FeS.

The reaction of sulfur removal using ferric oxide[9] adsorption[12] is:

$$H_2S\ (g) + \frac{1}{3} Fe_2O_3\ (s) \rightarrow \frac{1}{3} Fe_2S_3\ (s) + H_2O\ (l) \qquad (2.11)$$

ΔH= -22 kJ (at 25°C and 1 atm)

The Fe_2O_3 can be regenerated by interact with oxygen or air. After the process, sulfide element can clog in pores of adsorbent, therefore, adsorption performance will decrease after regeneration process. Moreover, Fe_2O_3 can be decompose into FeS_2 or Fe_8S_9 which the regeneration reaction is:

$$\frac{1}{3} Fe_2S_3\ (s) + \frac{1}{2} O_2\ (g) \rightarrow \frac{1}{3} Fe_2O_3\ (s) + S\ (s) \qquad (2.12)$$

ΔH= -198 kJ (at 25°C and 1 atm)

2.3.2 Chemical equilibrium

The chemical reaction equilibrium occurs when the reversible reaction proceeds in forward and backward equally. Most of the reactions could theoretically shift backward in closed system, however, this might not happen if the reaction is more like to shift forward to the product side. In this study, the reaction between Fe_2O_3 and H_2S is the reversible reaction since the reaction is operated in the closed batch system. However, the reaction species could be considered by the chemical reaction rate constant (K_C).

The value of K_c in this reaction can find by using relationship:

$$K_c = \frac{[H_2S][Fe_2O_3]^{1/3}}{[Fe_2S_3]^{1/3}} \qquad (2.13)$$

And it also find by using relationship below:

$$K_c \equiv \exp\left(\frac{-\Delta G^\circ}{RT}\right) \qquad (2.14)$$

Where,

$$\Delta G^\circ = \sum_i \nu_i \Delta G^\circ_i$$

$$= \frac{1}{3}(-166900) + (-237129) - (-33560) - \frac{1}{3}(-742200)$$

$$= -11802.3 \text{ J/mol}$$

Where, $\Delta G^\circ_{H_2S}$ (g) = -33560 J/mol

$\Delta G^\circ_{Fe_2O_3}$(s) = -742200 J/mol

$\Delta G^\circ_{Fe_2S_3}$(s) = -166900 J/mol

$\Delta G^\circ_{H_2O}$ (l) = -237129 J/mol

From equation (2.14):

$$K_c \equiv \exp\left(\frac{-\Delta G^\circ}{RT}\right)$$

$$K_c \equiv \exp\left(-\frac{(-11802.333)}{8.314(298.15)}\right) \equiv 116.9$$

The value of K_c shows that the reaction is not strongly forward reaction. Therefore, amount of each component for reaching equilibrium state can find according this equilibrium constant value.

2.4 Introduction of Foam

The polymer foam is a material that has a small cellular materials distributed in plastic. Polymer become to foam structure by gas dissolving and releasing in structure. The types of foam are rigid foam, semi-rigid foam, and flexible foam. An advantage of foam property is density, mechanical strength, insulation and etc. Polymeric foams can absorb a lot of energy. It can apply in packaging and cushioning applications. In addition, the small amount of solid polymer is needed in the foaming process. Therefore, manufacturing saves the cost of production. Polymeric foams are lightweight (densities ranging between 1.6 to as high as 960 kg/m^3) and high strength. The other properties of the polymeric foam are rapidly replaced other similar properties material. Foam structure can be classified into two types by using bubble or cell structure, open and close cell. First, open cell foam structure is gas inside the structure and can be move to another cell as show in Figure 2.5 (a). Second, close cell foam structure is that gas is trapped inside the cell and cannot pass through to another cell as show in Figure 2.5 (b).

(a) (b)

Figure 2.5 Internal structure of open cell foam (a) and open cell foam (b)[13]

2.4.1 Bubble Formation

Firstly, polymer is saturated by gas then instability is activated and applied into the system to generate a nucleus. Nucleation can be classified into two types which discuss as below.

1. **Homogeneous Nucleation**

Gas is dissolved into polymer and become homogeneous solution. Then, instability is activated and the system become phase separation that nuclei is generated. Homogeneous nucleation is illustrated and shown in Figure 2.6.

2. **Heterogeneous Nucleation**

The nuclei of this type are derived by the nucleating agents or impurities. They use to decrease surface tension between polymers and nuclei. Therefore the nuclei occurs between the polymer and the nuclei, nucleation site which, show in figure 2.6

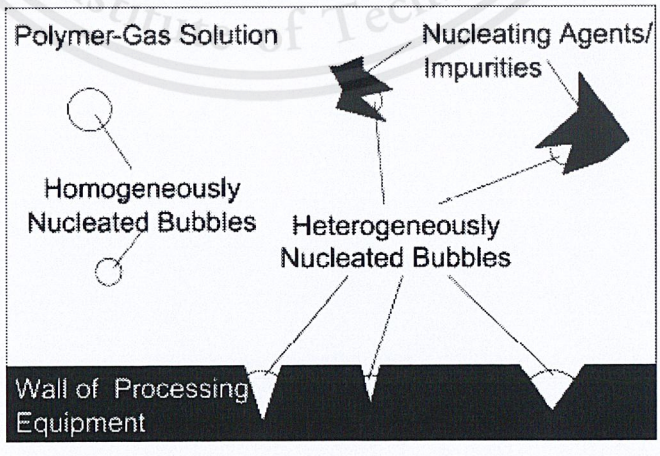


Figure 2.6 Homogeneous nucleation and heterogeneous nucleation in bubbles[14]

2.4.2 Bubble Growth

As dissolved gas become gas phase, its volume increases thus gas concentration inside nuclei reduce. It gain concentration driving force or concentration gradient as shown in Figure 2.7. Therefore, gas diffuse into the bubble and then the nuclei are enlarged.

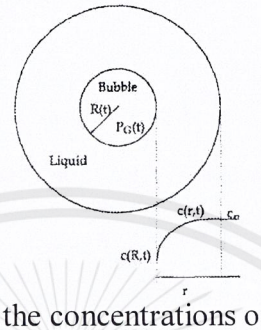


Figure 2.7 Relation between the concentrations of gas and radius of bubble[14]

2.4.3 Bubble Stability

The stability of gas bubbles as the final step of the process foaming. In this process, the size and bubble of foam are stable. Then, the dissolved gas escape to the atmosphere and at the time air enter into the foam. Therefore, the equilibrium is completely air into the foam and foam inside a pressure equal to atmospheric pressure.

2.4.4 Blowing Agent

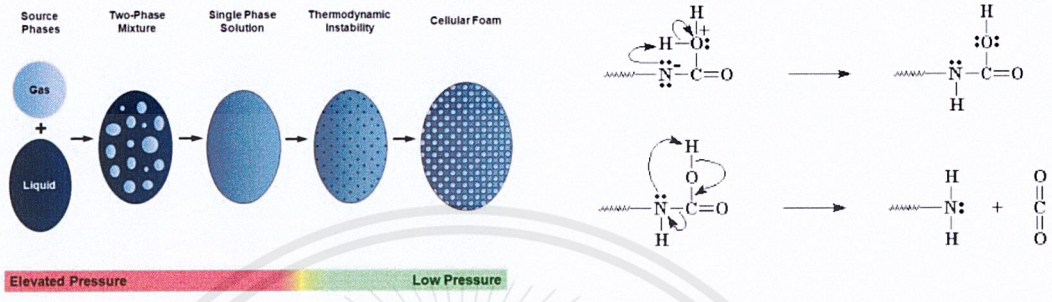
Generally, foaming process involve two methods which including of batch foaming process and continuous foaming process. Foaming structure can be involved by two type of blowing agents, physical and chemical blowing agents.

1. **Physical blowing agent**

Physical blowing agent is gas or volatile substance that can be dissolved in polymer matrix and become homogeneous solution such as carbon dioxide, nitrogen, methane, propane, butane. After gas dissolved in polymer matrix, the system is induced to thermodynamic instability by increasing temperature or decreasing pressure to activate phase separation mechanism. This mechanism is represented in Figure 2.8(a). Due to almost physical blowing agents are gas phase, transport phenomena is the first priority to concern and select before making a foam.

2. Chemical blowing agent

Chemical blowing agent is chemical substance that thermally decompose its structure or react to release gas into polymer matrix. An example of chemical blowing agent that decompose it structure and release gas (CO_2) is shown in Figure 2.8 (b).



(a) (b)

Figure 2.8 (a) foam formation by using physical blowing agent[15] and (b) decomposition structure of chemical reaction

2.5 Diffusivity in polymer foam and time of adsorption

Typical gases in polymer system are nitrogen, oxygen, helium, carbon dioxide, sulfur dioxide, methane and etc. They are diffused by mass transfer at relatively low pressure or equivalently low activities. The gases can be called "permanent gases". The model law to find diffusivity of gases is Lennard-Jones equation.

$$\Phi(r)=4\varepsilon\left[\left(\frac{\sigma}{r}\right)^{12}-\left(\frac{\sigma}{r}\right)^{6}\right] \tag{2.15}$$

Where $\Phi(r)$ is the intermolecular energy of two molecules r distance apart, ε is the potential energy constant and σ is the potential length constant. Note ε and σ are called Lennard-Jones scaling factors and σ can also call collision diameter of the molecule. Division of ε by Boltzmann constant (k) is Lennard-Jones temperature ε/k. The simple gases properties can be found in Appendix F.1 from Svehla et al (1962).

The relation between the diffusion coefficient and the temperature is expressed by the Arrhenius-type equation.

$$D=D_0\exp\left(-E_D/R_gT\right) \tag{2.16}$$

Where D_0 and E_D are constants for the particular gas-polymer system. D_0 is the pre-exponential factor for D. E_D is the activated energy of diffusion and R_g is universal gas constant.

The pre-exponential factor (D_0) and activated energy of diffusion (E_D) values can find from this equations.

$$10^{-3}\frac{E_D}{R_g} = \left(\frac{\sigma_{H_2S}}{\sigma_{N_2}}\right)^2 \left[7.5\text{-}2.5\times10^{-4}\left(T_g\text{-}298\right)^{3/2}\right] \tag{2.17}$$

$$\log D_0 \cong \frac{E_D \times 10^{-3}}{R_g} \text{-}5.0\pm0.8 \tag{2.18}$$

Where σ is collision diameter of the molecule in table appendix F.1, R_g is universal gas constant and T_g is glass transition temperature.

The time of adsorption in PE foam (assume adsorption in a slab)

$$t = \frac{b^2}{D} \tag{2.19}$$

Where; t = The time of adsorption
b = (The thickness of cell wall)/2
D = Diffusivity

2.6 Expansion Ratio, Ø

The expansion ratio is employed to define an order of volume expansion of polymer foam relate it original polymer. As definition, expansion ratio is the ratio of polymeric foam volume (V_F) and polymer volume (V_P) as present in Equation 2.20. Moreover, it is difficult to measure volume of an irregular shape so the equation have been simplified into a function density.

$$\emptyset = \frac{V_F}{V_P} = \frac{(\tilde{m}_p+\tilde{m}_g)/\rho_F}{\tilde{m}_F/\rho_P} \tag{2.20}$$

When $\tilde{m}_p$ is mass of the polymer (g)
$\tilde{m}_g$ is mass of gas (g)
ρ_F is the density of foam (g/cm^3)
ρ_P is the density of polymer (g/cm^3)

The weight of gas is very low. Therefore the expansion ratio increase follows the equation.

$$\emptyset = {}^{\rho_P}/_{\rho_F} \tag{2.21}$$

When $\emptyset$ is expansion ratio

ρ_F is the density of foam (g/cm^3)

ρ_P is the density of polymer (g/cm^3)

CHAPTER III
RESEARCH METHODOLOGY

3.1 Materials and equipment

Materials

1. Low-density polyethylene (LDPE) grade C150Y with melt flow index (MFI) of 5.24 g/10 minutes was supplied from Petronas Chemicals Limited Company.

2. The ferric (III) oxide powder (Fe_2O_3) grade 96% purity was supplied from Henan Premtec Enterprise Company.

3. Ferric (II) sulfide (FeS) for prepare hydrogen sulfide gas production was supplied from Gammaco Company.

4. Hydrochloric acid (HCl) grade is 37%v/v was diluted to 0.0002 mol/dm^3 and 1 mol/dm^3

Equipment

1. Twin screw extruder (Kurimoto Ltd.)
   KRC KNEADER
   The rotational speed between 200 rpm and temperature 200 °C

2. Density testing machine (ALFA Mirage CO, LTD)
   The model MD-200S
   The resolution 0.001g/cm^3
   Maximum measuring range 0 – 300 g

3. Physical polymeric foam reactor
   Maximum pressure range 0 - 4000 psi

4. Silicon oil bath with agitation and heater (Fortune scientific CO, LTD)

5. The pH-indicator strips (Merck KGaA, Germany)
   The pH measuring between 0 – 14

6. Gas data meter detector (GFM435 Series)

Equipment for compounding process

1. Turbo mixer batch size 50 kg

Figure 3.1 Turbo mixer

2. Twin screw extruder for compounding process

Figure 3.2 Twin screw extruder

Equipment for foaming process

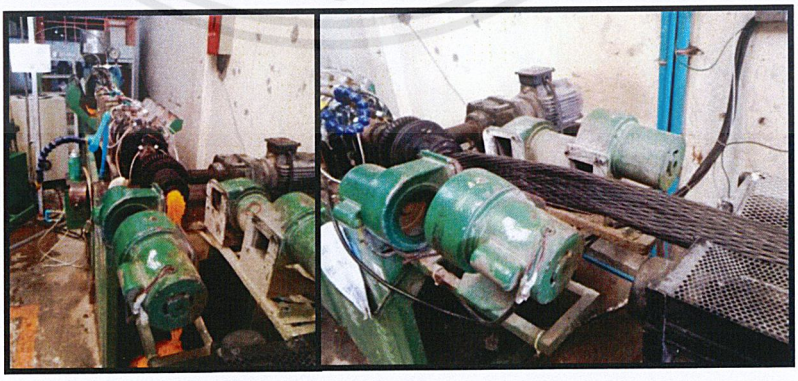


Figure 3.3 Continuous foaming process

3.2 Experimental procedures

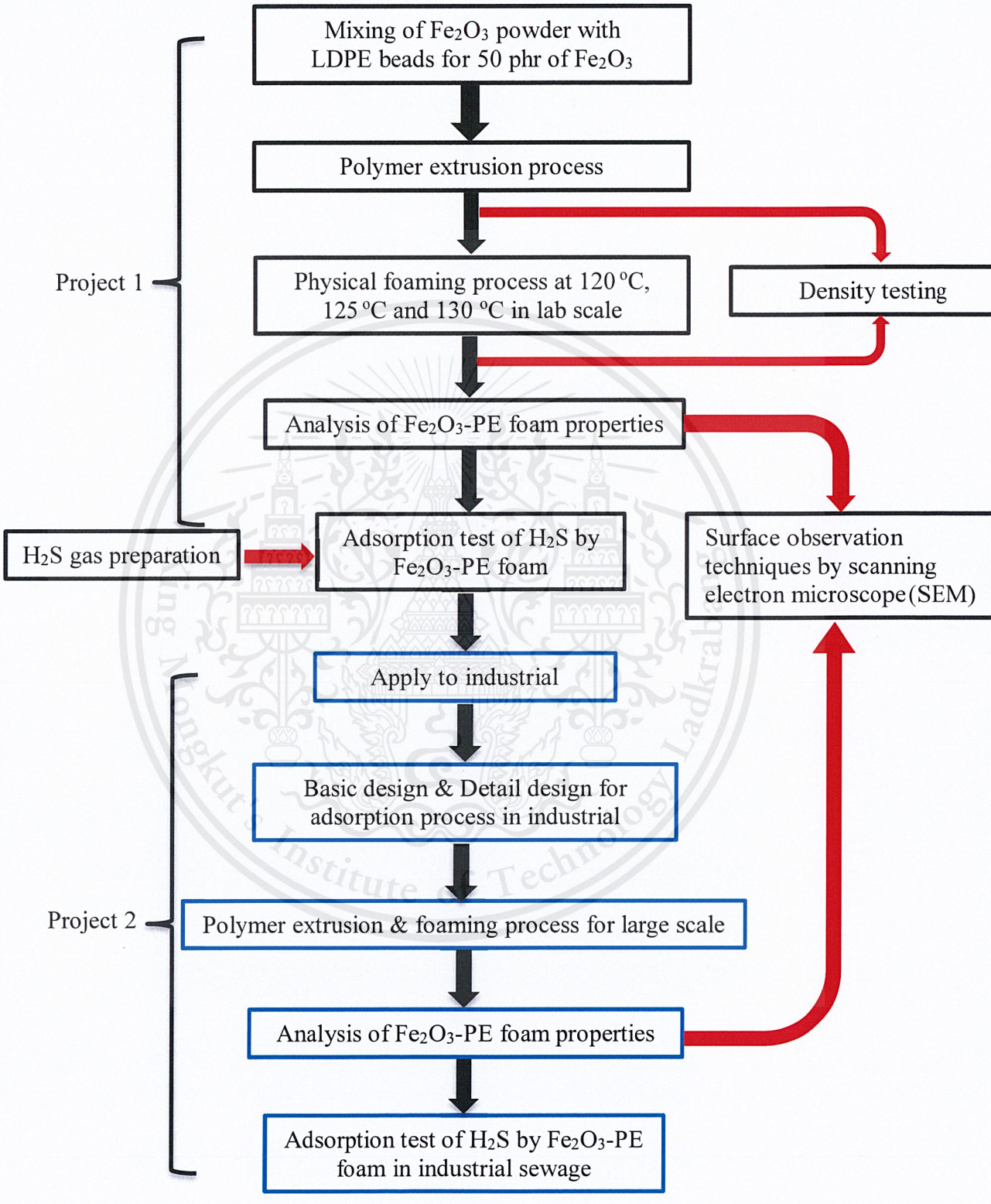


Figure 3.4 Experimental procedures

3.2.1 Mixing and extrusion process

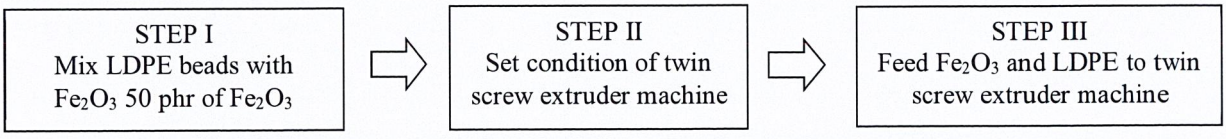
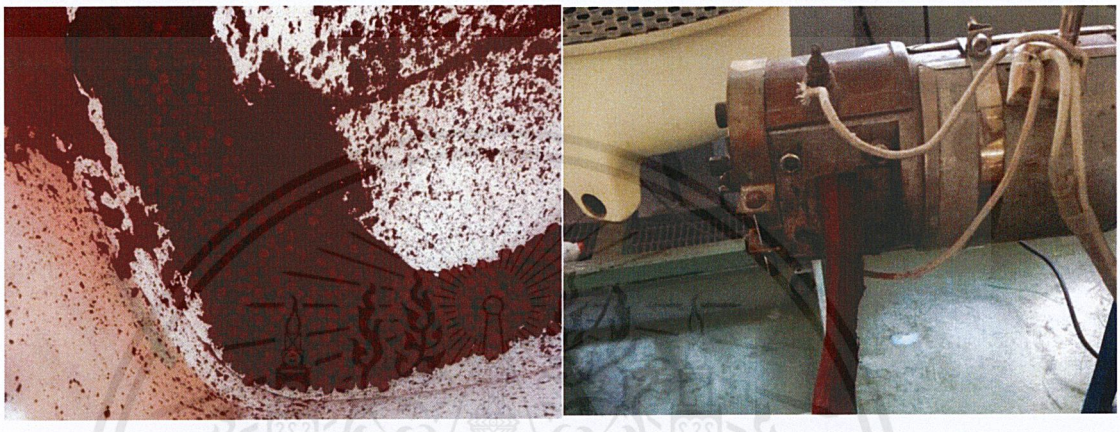


Figure 3.5 Procedures of mixing and extrusion process



(a) (b)

Figure 3.6 (a) LDPE beads and Fe_2O_3 powder compounding in a bucket and (b) twin screw extruder

The mixing ratio between LDPE beads and Fe_2O_3 powder at 50 phr of Fe_2O_3 to form Fe_2O_3-PE compound. Then, they were loaded to twin screw extruder machine. Twin screw extruder machine operates at 200°C and rotational speed 200 ppm. After the extrusion process, molding must be cleaned by LDPE. The Fe_2O_3-PE must be manually controlled as thin as possible because it can use for physical polymer foaming process.

3.2.2 Foaming process

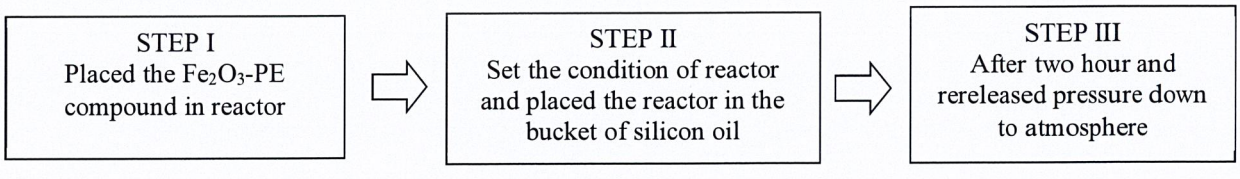
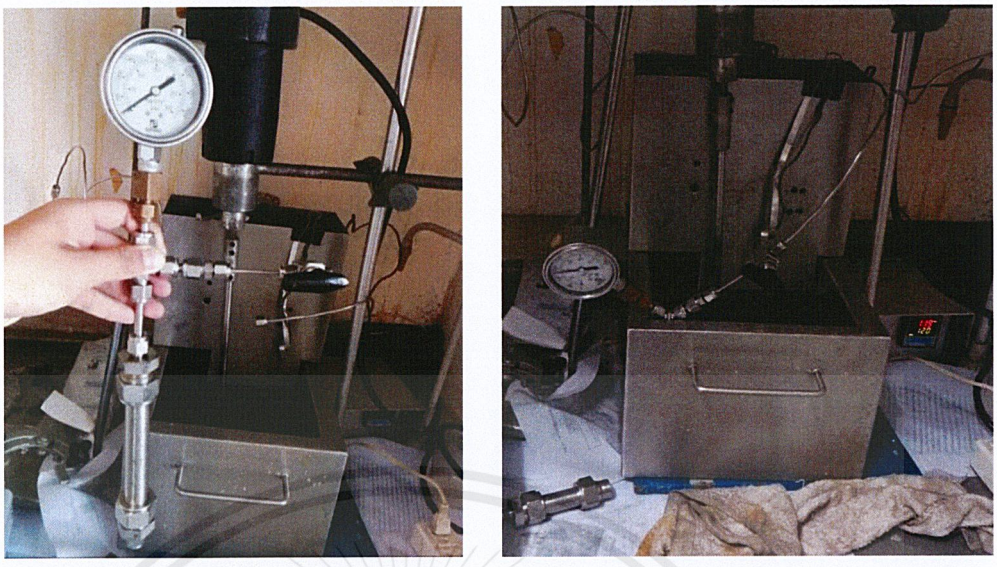


Figure 3.7 Procedures of foaming process



(a) (b)

Figure 3.8 (a) Physical polymeric foam reactor and
(b) Silicon oil bath with agitation and heater

The Fe_2O_3-PE compound was cut as small pieces for placing into the high pressure vessel that shows in figure 3.8(a). It was filled with carbon dioxide gas at pressure of 1500 psi and temperature of 120 °C, 125 °C, and 130 °C. To excess diffusion time of carbon dioxide into polymer, the system was left for two hours after that rapidly reducing pressure into atmospheric within 3-5 s to make a foam.

3.2.3 Hydrogen sulfide gas preparation

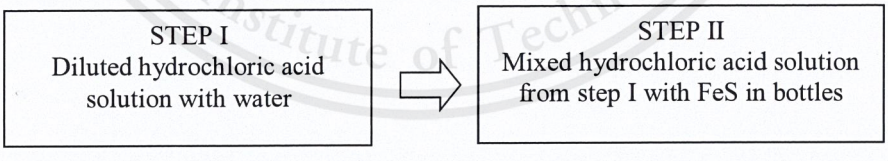


Figure 3.9 Procedures of hydrogen sulfide gas preparation

There are many reactions produce to hydrogen sulfide gas. This case, hydrogen sulfide gas is formed by the hydrochloric acid solution and excess ferric (II) sulfide (FeS). H_2S is prepared by calculating stoichiometry number of FeS and HCl according to the reaction. In addition, the reaction has to low in order to avoid solubility effect of hydrochloric acid solution in the aqueous phase. The concentration of the hydrochloric acid solution are 0.0002 mol/dm^3 (10 ml). It was prepared by dilute hydrochloric acid solution 37%v/v with water.

3.2.4 Fe_2O_3-PE testing of properties

1. Density testing

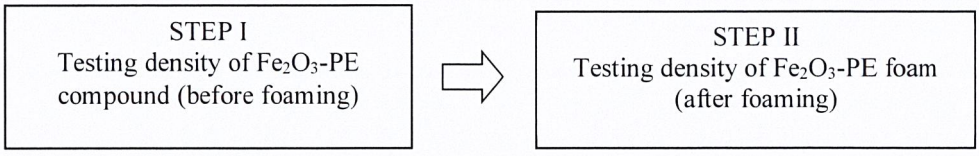


Figure 3.10 Procedures of density testing

The density of Fe_2O_3-PE values for ratio 50 phr of Fe_2O_3. It was assumed perfect mixing from the extrusion process. The density of the Fe_2O_3-PE compound and foam were measured by densimeter machine. The density values of Fe_2O_3-PE compound (SG>1) higher than Fe_2O_3-PE foam (SG<1) because the foam is expansion ratio after foaming processes.

2. Scanning electron microscope (SEM) testing

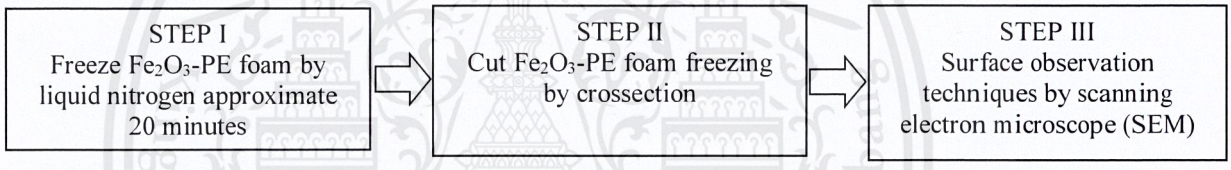


Figure 3.11 Procedures of scanning electron microscope (SEM) testing

Scanning electron microscope (SEM) was used to scanning surface observation inside the Fe_2O_3-PE foam. In the work, EVO® HD model in SEM from Carl Zeiss was applied. Delivering a groundbreaking increase in resolution over conventional SEM, the EVO® HD introduces high definition to electron microscopy.

3.2.5 Adsorption test of H_2S by Fe_2O_3-PE foam (pH indicator)

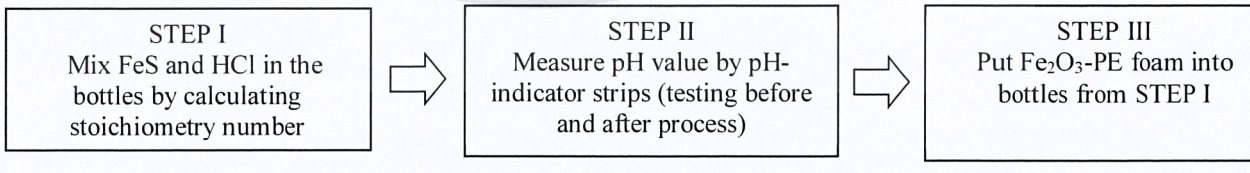
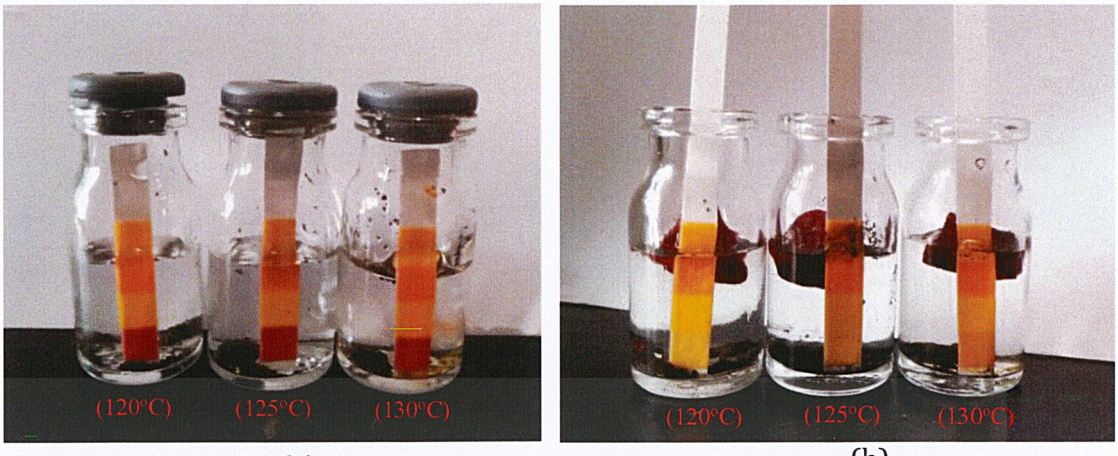


Figure 3.12 Procedures of adsorption test of H_2S by Fe_2O_3-PE foam



(a) (b)

Figure 3.13 (a) Measure pH value by pH-indicator strips before process and (b) Measure pH value by pH-indicator strips after process

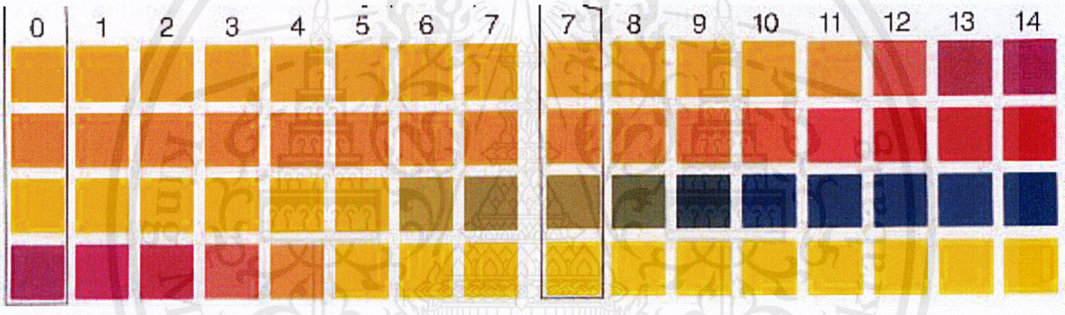


Figure 3.14 The pH indicator strips

The adsorption test result of H_2S by Fe_2O_3-PE foam at different conditions (temperature of 120 °C, 125 °C, and 130 °C) is shown in figure 3.14 HCl was mixed with excess FeS, hence the mixture contains only H_2S and the excess FeS. The mixture were left over two days for the reaction to occur and reach the equilibrium state. The remaining H_2S after the reaction was detected and measured by pH indicator strips. This testing is a basic test to confirm that Fe_2O_3-PE can adsorb H_2

3.3 Scaling up Fe_2O_3-PE foam (pilot scale)

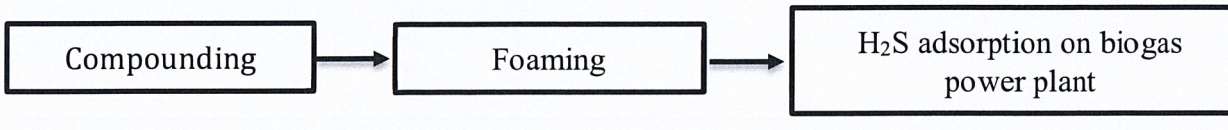


Figure 3.15 Procedures of Fe_2O_3-PE foam for industrial

The scale-up procedure for the industrial scale is similar with the laboratory scale, which contains compounding process, foaming and H_2S adsorption described as follows.

3.3.1 Compounding

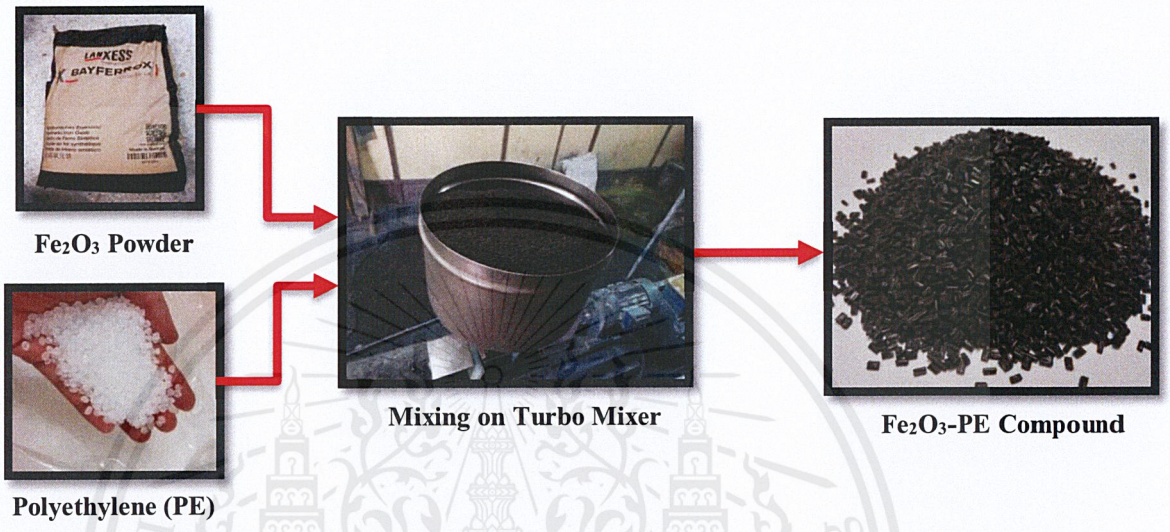


Figure 3.16 Procedures of compounding

The compounding process starting with mixing Fe_2O_3 powder and polyethylene pellets (PE) in the turbo mixer with the ratio of 60 phr of Fe_2O_3 to aim well mixed mixture. The mixture then goes through the twin screw extruder to make the polymer compound for the foaming process.

3.3.2 Foaming

Figure 3.17 Procedures of foaming

After achieving the Fe_2O_3-PE compound, it would be foamed using iso-butane gas as the physical blowing agent with the flow rate of 8 L/h and the pressure of 100 bar. This process is operated at the temperature of 125°C to melt the Fe_2O_3-PE compound and foamed.

3.3.3 Hydrogen sulfide gas preparation

This case, hydrogen sulfide gas is formed by the hydrochloric acid solution and excess ferric (II) sulfide (FeS). The concentration of the hydrochloric acid solution are 1 mol/dm^3 (40 ml). It was prepared by dilute hydrochloric acid solution 37%v/v.

3.3.4 Fe_2O_3-PE testing of properties

1. Density testing
2. Scanning electron microscope (SEM) testing

Note: Repeat the procedure in topic 3.2.4

3.3.5 Adsorption test of H_2S by Fe_2O_3-PE foam (Gas data meter detector)

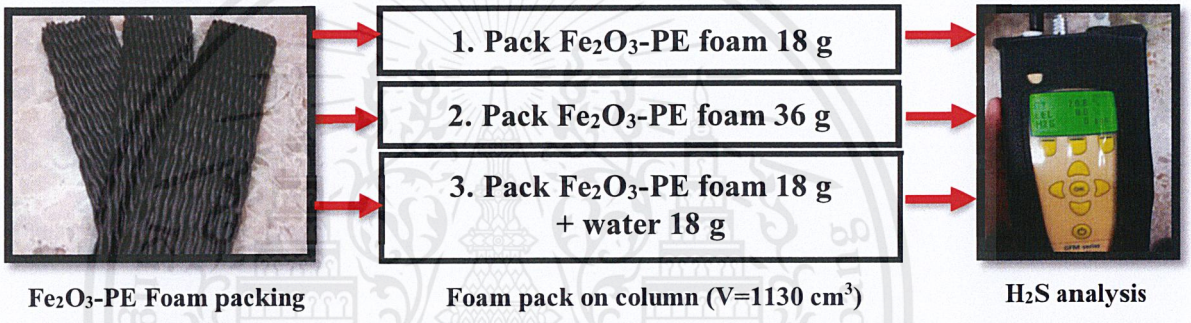


Figure 3.18 Procedures of foaming H_2S adsorption testing

The efficiency of the Fe_2O_3- PE foam is tested by packing the foam in the column to trap H_2S gas after entering the process. The H_2S amount is then measured by the gas data meter detector.

3.4 Apply to industrial (Bio gas power plant)

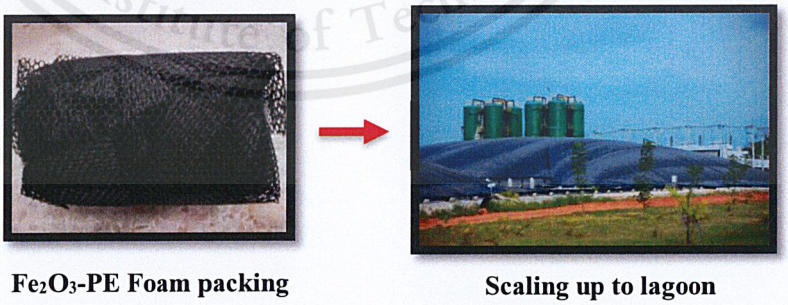


Figure 3.19 Scaling up to large lagoon

After achieved the adsorption data at header, the next step is to scaling up. The experimental data from the lab scale will be used to scaling up the foam and then test the H_2S adsorption performance of the foam in the large lagoon.

CHAPTER IV
RESULTS AND DISCUSSION

4.1 Fe_2O_3-PE foam testing (temperature effect)

4.1.1 Density and expansion

Table 4.1 Density values of extruded polymer and Fe_2O_3-PE compounded foam

Polymer compound		Fe_2O_3-PE of foam		
Fe_2O_3 powder/LDPE beads mixing ratio	Density (g/cm^3)	Temperature for foaming process (°C)	Density (g/cm3)	Expansion ratio
50 phr of Fe_2O_3	1.297	120	0.176	7.37
50 phr of Fe_2O_3	1.297	125	0.159	8.20
50 phr of Fe_2O_3	1.297	130	0.530	2.45

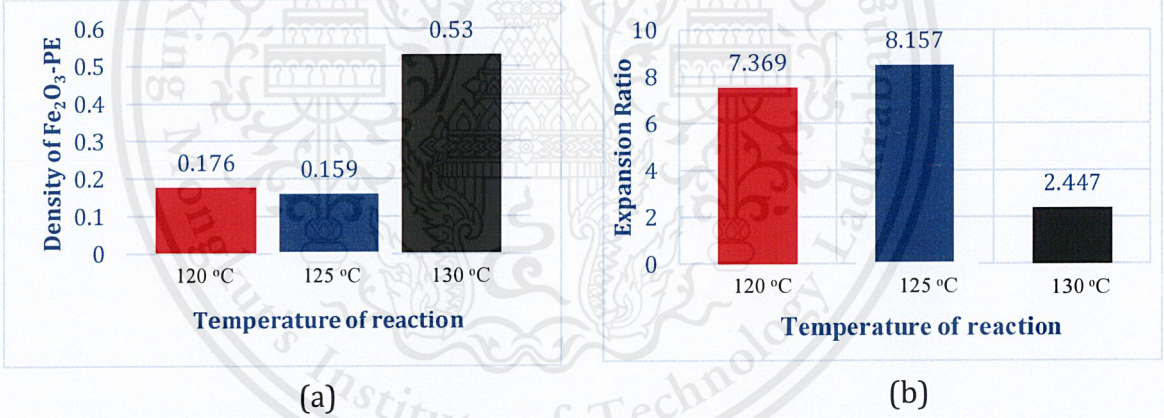


Figure 4.1 (a) Relation between density of Fe_2O_3-PE and temperature of reaction and (b) Expansion ratio of Fe_2O_3-PE foam and temperature of reaction

The extrusion process was assumed perfect mixing. The densities of polymer compounds were measured by density testing machine. The density values of each polymers compound can assume for non-gas trapping inside. Polymers compounds are transformed to Fe_2O_3-PE foam, the density value was decreased to the range of 0.176 - 0.530. Therefore it can ensure of polymer foam achievement and have the ability for floating. They obtained an expansion ratio about 2.4 - 8.2 times. The suitable temperature reaction is 125°C because Fe_2O_3-PE of foam is high expansion ratio and low density values. Therefore, the reaction at 125°C is used for next steps.

4.1.2 Scanning electron microscope (SEM)

Fe_2O_3-PE at the operating pressure of 1,500 psi and temperatures of 120 °C, 125 °C, and 130 °C.

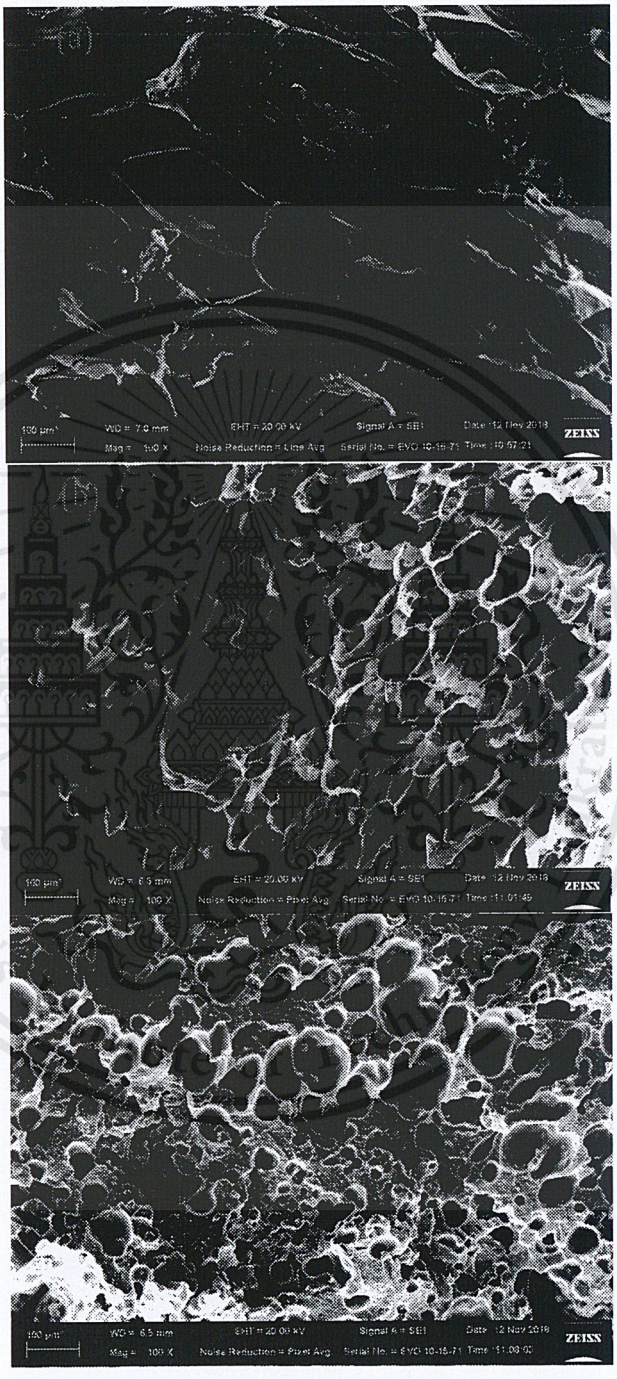


Figure 4.2 50 phr of Fe_2O_3-PE compounded foam
at temperature of (a)120°C, (b)125°C and (c)130°C at magnitude 100X

1. Fe_2O_3-PE foam at temperature of 120 °C and pressure of 1,500 psi.

The expansion ratio of Fe_2O_3-PE foam at this condition according to Table 4.1 is high which can imply for large pores gain. There is a high distribution of pores and the pore size is non-perfect uniformity which shown in Figure 4.2. The pore size of the Fe_2O_3-PE foam is approximately 100-300 micrometers. It is classified as microcellular polymer foam type. Therefore, the gas could easily flow through the Fe_2O_3-PE foam even in the low gas pressure condition. However, this polymer foam is not the full open-cell type which makes some area inside the foam are unable for the reaction of gas. The particles of Fe_2O_3-PE have well distribution on Fe_2O_3-PE foam. The result at this condition gives the largest pore size of Fe_2O_3-PE foam.

2. Fe_2O_3-PE foam at temperature of 125 °C and pressure of 1,500 psi.

The Fe_2O_3-PE foam achieved from this condition has similar expansion ratio with the previous condition (temperature of 120 °C and pressure of 1,500 psi) show in Table 4.1. The pore size of the Fe_2O_3-PE foam is approximately 20-200 micrometers. It is classified as macrocellular polymer foam type. The gas could also easily flow through the Fe_2O_3-PE foam even in the low gas pressure condition. However, this polymer foam is not the full open-cell type which make some area inside the foam are unable for the reaction of gas. The particles of Fe_2O_3-PE have well distribution on Fe_2O_3-PE foam. The result at this condition shows the highest surface area of Fe_2O_3-PE foam because they have many bubbles inside the foam.

3. Fe_2O_3-PE foam at temperature of 130 °C and pressure of 1,500 psi.

The Fe_2O_3-PE foam achieved from this condition has different expansion ratio with other condition. During the change of condition by adjusting the temperature, the foam melt due to the increase of temperature. Therefore, the gas could hardly flow through the Fe_2O_3-PE foam even in the low gas pressure condition. The Fe_2O_3-PE foam at this condition contains low surface area and low pore size. On the other hand, the density of the foam is high. This condition founds to be the worst condition comparing to the other two conditions.

4.2 Estimate diffusion of H_2S in PE foam estimation (temperature effect)

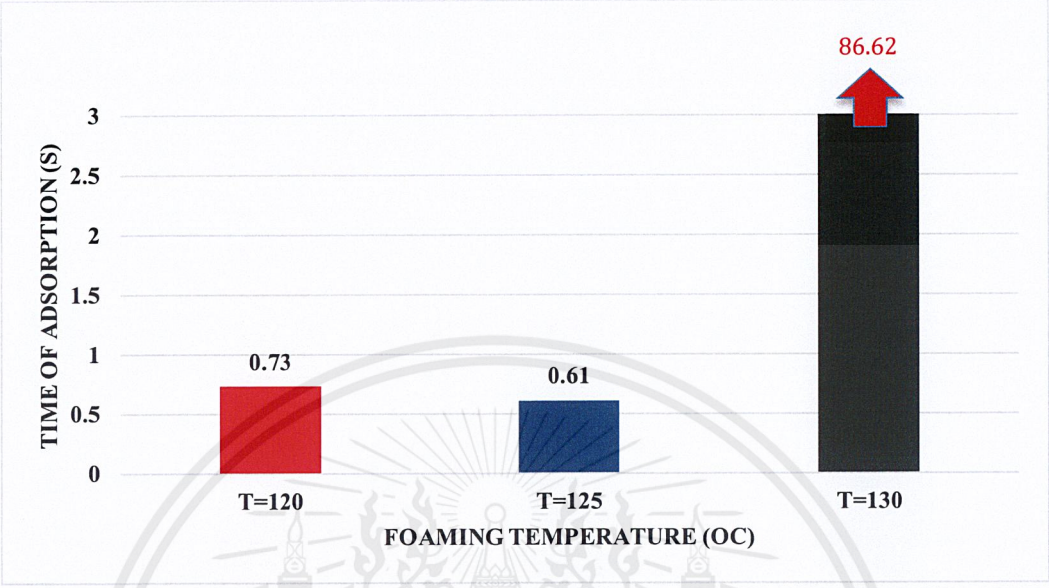


Figure 4.3 Relation between foaming temperature and the time of adsorption

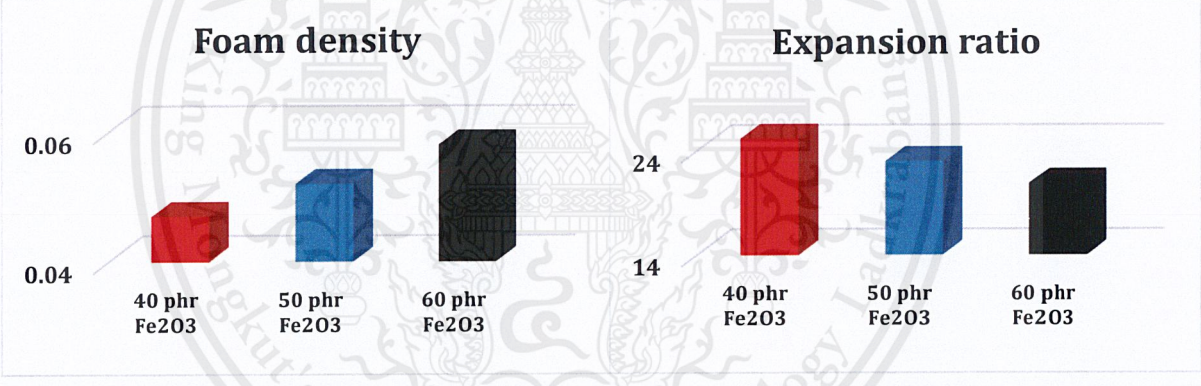
The physical foaming process was assumed perfect mixing. The time of adsorption of H_2S to Fe_2O_3-PE foam were calculated by diffusion in a slab (assume gas diffusion hollow sphere same diffusion in a slab). The time of adsorption of each temperature conditions (temperature of 120°C, 125°C, and 130°C) are ranges of 0.6 – 86.7 s. The foaming temperature each conditions (temperature of 120 °C and 125 °C) were close value (time of diffusion of 0.61 and 0.73 day). Therefore, it can ensure that polymer foam can be achieve and have the ability for adsorption. The suitable temperature reaction is 125°C because Fe_2O_3-PE of foam is less time of diffusion and good morphology of foam.

4.3 Apply to industrial (pilot scale)

4.3.1 Density and expansion

Table 4.2 Density values of extruded polymer and Fe_2O_3-PE compounded foam

Polymer compound		Fe_2O_3-PE of foam	
Fe_2O_3 powder/LDPE beads mixing ratio	Density (g/cm^3)	Density (g/cm3)	Expansion ratio
40 phr of Fe_2O_3	1.183	0.047	25.17
50 phr of Fe_2O_3	1.196	0.052	23.00
60 phr of Fe_2O_3	1.210	0.058	20.86



(a) Foam density (b) Expansion ratio

Figure 4.4 (a) The foam density of 40, 50 and 60 phr of Fe_2O_3
(b) Expansion ratio of 40, 50 and 60 phr of Fe_2O_3

Figure 4.4 shows the expansion ratio of the foam with different Fe_2O_3 ratio. It is observed that the highest expansion ratio could be achieved at the ratio of 40 phr of Fe_2O_3, where the 60 phr of Fe_2O_3 foam has the lowest expansion ratio. However, the Figure 4.4 shows no difference of the foam density at approximately 0.058 g/cm^3, which makes the foam well - floating on the water. Moreover, the foam with 60 phr of Fe_2O_3 has the most Fe_2O_3 loading amount which means it contains the most amount of H_2S adsorbent and leads to the saving of PE used. Therefore, the foam with 60 phr of Fe_2O_3 is chosen.

4.3.2 Scanning electron microscope (SEM)

1. **40 phr of Fe_2O_3**

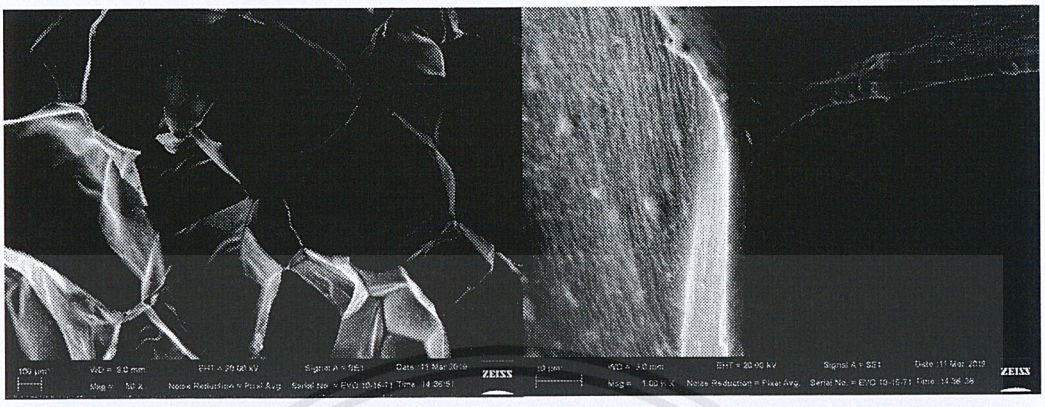


Figure 4.5 40 phr of of Fe_2O_3-PE foam at magnitude 50X with 1000X

2. **50 phr of Fe_2O_3**

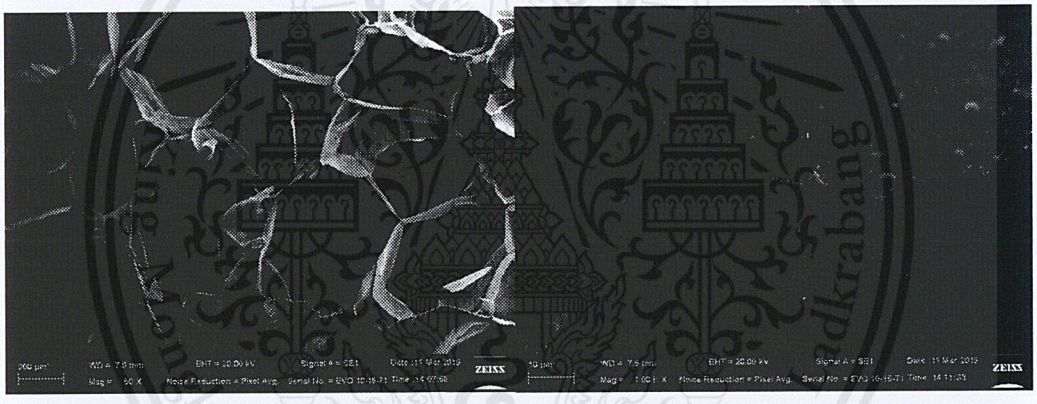


Figure 4.6 50 phr of of Fe_2O_3-PE foam at magnitude 50X with 1000X

3. **60 phr of Fe_2O_3**

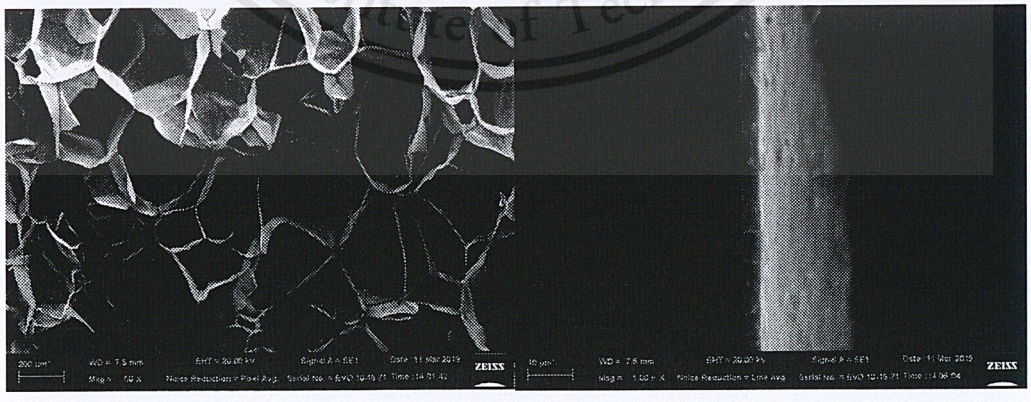


Figure 4.7 60 phr of of Fe_2O_3-PE foam at magnitude 50X with 1000X

The effects of temperature to the morphology of the foam is investigated in the lab scale, where the suitable temperature and pressure is achieved. However, the pilot-scale production is the continuous process, where the mixing ratio of the compound that suits with the equipment is needed to be investigated. Thus, the compound with the mixing ratio of 40, 50 and 60 phr of Fe_2O_3 is prepared. The result shows that all of them could be compounded and foamed in the continuous process,

4.3.3 Estimate diffusion of H_2S in PE foam estimation

Table 4.3 Thin of wall of Fe_2O_3-PE foam and the time of adsorption in PE foam

Fe_2O_3-PE of foam	Thin of wall (μm)	The time of diffusion in PE foam (estimated by Arrhenius-type equation)
40 phr of Fe_2O_3	3.48	0.93 s
50 phr of Fe_2O_3	3.62	1.00 s
60 phr of Fe_2O_3	4.41	1.49 s

Table 4.3 shows the time of diffusion in PE foam is estimated from Equation 2.17. The result shows that the diffusion of H_2S through the cell wall and goes inside the polymer requires a few seconds, which is not the problem for the H_2S to diffuse and reacts with Fe_2O_3 that dispersed in the foam. This results also will be the primary data for designing the H_2S adsorption performance test in the following section.

The morphology of the foam with 40, 50 and 60 phr of Fe_2O_3 of shows the well dispersion of Fe_2O_3 powder in the polymer and uniform bubble formation and dispersion. Moreover, it contains thin cell wall approximately 2-3 microns, which suitable for the gas diffusion through the cell wall, that the retention time could be estimated from the Arrhenius equation. The calculation result shows the diffusion time of gas through the cell wall of 40, 50 and 60 phr of foam approximately 0.93 seconds, 1.00 seconds and 1.49 seconds respectively.

The effects of the Fe_2O_3-PE mixing ratio to the morphology of the foam is investigated by SEM. Figure 4.5, 4.6 and 4.7 are the SEM pictures of the foam with 40, 50 and 60 phr of Fe_2O_3, which show well morphology of foam. Therefore, the next criteria that would be discussed is the amount of Fe_2O_3 loading since the amount of Fe_2O_3 affects directly to the amount of H_2S that could be adsorbed. The higher amount of Fe_2O_3 loaded could lead to the higher amount of H_2S adsorbed. However, too much amount of Fe_2O_3 would make the mixture unable to be compounded and foamed. Thus, the ratio of 60 phr of Fe_2O_3 is chosen since the mixture with more than 60 phr of Fe_2O_3 could not be compounded and pelletized into the pellets by twin screw extruder, which is unable to be foamed in the continuous process.

4.3.4 H_2S adsorption testing (batch column)

The hydrogen sulfide adsorption performance of Fe_2O_3-PE foam is investigated by set up the column then packed the foam inside the column which bulk density are 0.015 g/cm^3, 0.030 g/cm^3 and 0.015 g/cm^3 with the presence of water. The hydrogen sulfide concentration is measured at 0, 15, 30, 45, 60, 90, 120, 150 and 180 min, which shows in Tables 4.4, 4.5 and 4.6 respectively. The data could be plotted as show in figure 4.8.

Table 4.4 Bulk density of foam is 0.015 g/cm^3

Reaction time (min)	H_2S concentration (ppm)
0	>5000
15	>5000
30	>5000
45	>5000
60	3880
90	1770
120	1085
150	860
180	580

Table 4.5 Bulk density of foam is 0.030 g/cm^3

Reaction time (min)	H_2S concentration (ppm)
0	>5000
15	>5000
30	>5000
45	4885
60	4145
90	1845
120	885
150	450
180	285

Table 4.6 Bulk density of foam is 0.015 g/cm^3 with the presence of water

Reaction time (min)	H_2S concentration (ppm)
0	>5000
15	>5000
30	>5000
45	4980
60	3945
90	2305
120	1995
150	1665
180	1445

Tables 4.4, 4.5 and 4.6 show the relationship between H_2S concentration (ppm) and the reaction time (min). However, there is the limitation of the gas data meter (the concentration over 5000 ppm could not be detected) leads to the lack of data at 0, 15, 30 and 45 min. Therefore, the data at that time should be estimated by the trend of the graph made from the data at 60, 90, 120, 150 and 180 min.

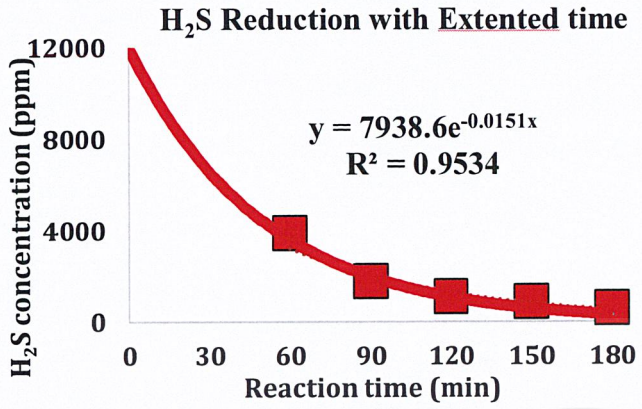
Table 4.6 shows foam packing at the wet condition. This effect occurs a foam which has a lot of water in the foam will affect to H_2S which react with H_2O to produce H_3O^+. The remain H_2S will react with foam, so the initial of H_2S will rapidly decreasing. Once the rate of reaction will slower than another due to H_2O is a product of this reaction which increasing product and decrease the forward rate of reaction.

The data achieved from the experiment is then plotted into a graph, which shows the exponential trend, thus the equation could be determined by the following.

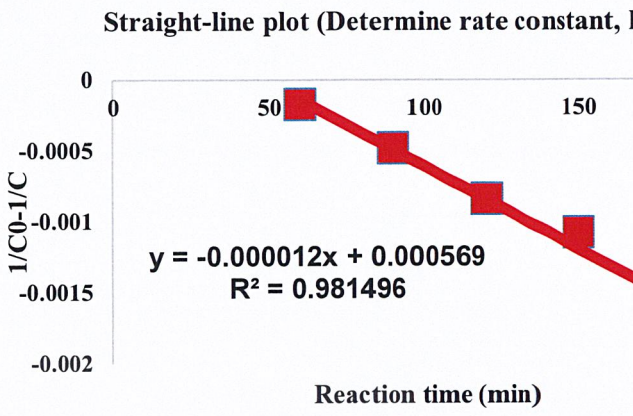
$$Y = Y_0e^{-mx} \quad (4.1)$$

$$C = C_0e^{-kt} \quad (4.2)$$

1. Bulk density of foam packing 0.015 g/cm^3

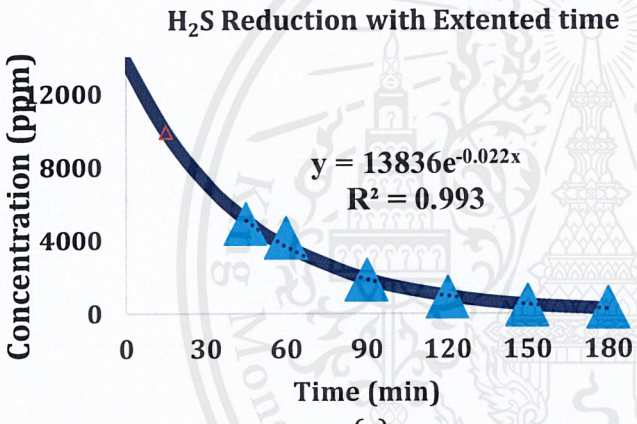


(a)

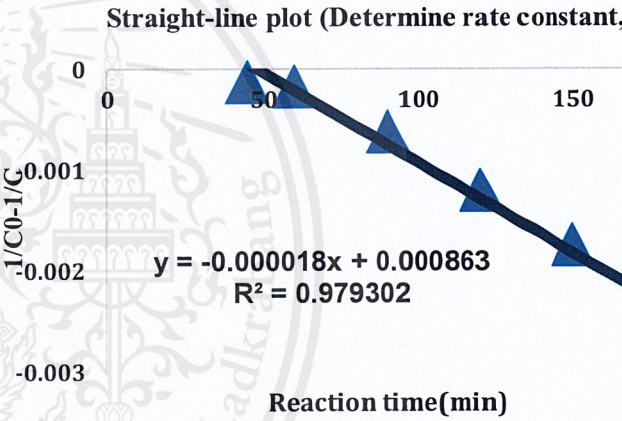


(b)

2. Bulk density of packing foam 0.015 g/cm^3

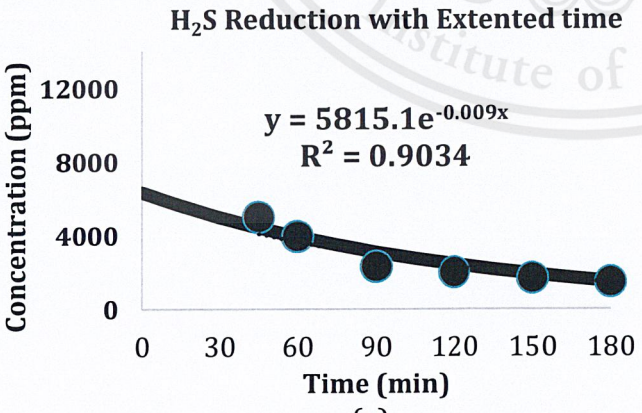


(c)

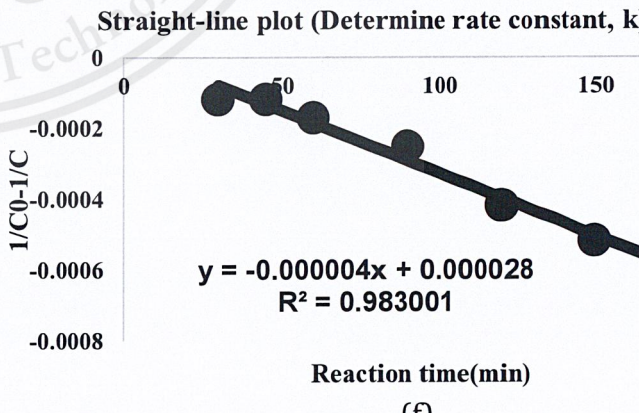


(d)

3. Bulk density of packing foam 0.015 g/cm^3 with the presence of water



(e)



(f)

Figure 4.8 (a,c,e) Relation between H_2S concentration and time
(b,d,f) Relation between $\ln C_0$-$\ln C$ and time

Figure 4.8 shows the trend of second-order reaction. Therefore, the reaction is assumed as the second – order reaction and could be explained by the Equation 4.3.

$$\ln C_0 - \ln C = kt \quad (4.3)$$

According to equation 4.3, the reaction rate constant (k) can be determined as showed in Table 4.7.

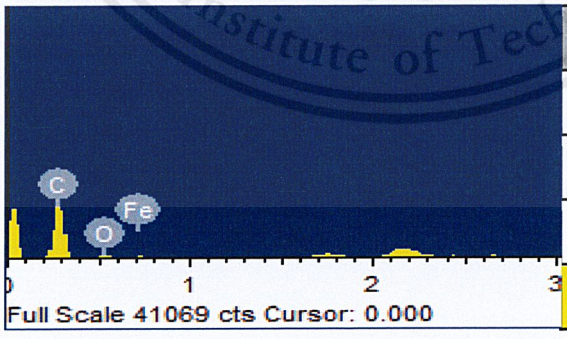
Table 4.7 Rate constant (k) calculated from the experiment

Bulk density of foam packing (g/cm^3)	Rate constant (k) (min^{-1})	R^2
0.015	1.2×10^{-5}	0.98
0.030	1.8×10^{-5}	0.98
0.015+water	4.0×10^{-6}	0.98

The reaction rate constant (k) that bulk density has 0.030 g/cm^3 is 1.8×10^{-6} min^{-1} which the highest of reaction rate constant (k). The bulk density is 0.015 g/cm^3 with the presence of water giving the lowest of reaction rate constant which is 4.0×10^{-6} g/cm^3. The reaction rate constant (k) value is then used for determine the time of adsorption, where the concentration over time can be set.

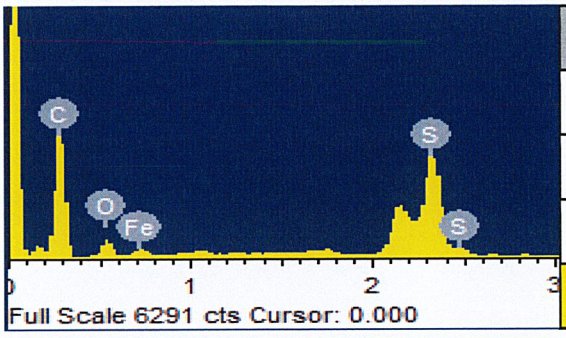
4.4 SEM-EDX analysis

According to the EDX spectrum analysis inside the foam



Element	Weight%
C	69.06
O	6.37
Fe	24.57
S	0.00

Figure 4.9 60 phr of Fe_2O_3-PE foam before adsorb H_2S



Element	Weight%
C	65.64
O	4.72
Fe	25.30
S	4.34

Figure 4.10 60 phr of Fe_2O_3-PE foam after adsorb H_2S

The experiment of H_2S adsorption by Fe_2O_3-PE foam can explain the reaction rate by the equation. However, in order to confirm that Fe_2O_3-PE foam could really adsorb H_2S, the comparison between Fe_2O_3-PE foam before and after H_2S adsorption is shown in Figure 4.9 and 4.10 respectively. The EDX pattern shows that the foam before H_2S adsorbing has no sulfur content, where the foam after H_2S adsorbing has sulfur content present in the EDX pattern, which confirmed that the H_2S adsorption is occurred.

CHAPTER V
CONCLUSION

5.1 Conclusion

In this present work, the adsorption of H_2S by the Fe_2O_3 – PE foam is studied, where the study has divided into the lab – scale section and the scale – up section for the operation in the industrial scale. In the lab – scale experiment, the suitable condition for the foaming process is investigated. The Fe_2O_3 – PE compound has been foamed under the temperature of 120°C, 125°C and 130°C with the pressure of 1,500 psi and CO_2 is used as the blowing agent. The foam achieved is then analyzed by SEM to study the morphology of the foam. The result shows that the morphology of the foam achieved under the temperature of 120 °C and 125 °C has the well dispersion of Fe_2O_3 and contains uniform bubble. Moreover, the cell wall thickness is approximately 3–5 microns, which is suitable for the H_2S capturing.

Before scaling up, the Fe_2O_3 – PE compound is prepared by the twin screw extruder, which the mixing ratio needs to be investigated. The Fe_2O_3 – PE mixing ratio of 40, 50 and 60 phr of Fe_2O_3 has been mixed, compounded and estimated its density by the densimeter. The compounded with different mixing ratio is then foamed in the continuous foaming process under the temperature of 125 °C with iso-butane as the blowing agent. The morphology of the foam is then investigated and estimated its expansion ratio. The study shows that all of the foam with different mixing ratio has similar cell wall thickness (3 – 5 μm) and the gas diffusion time through the cell wall is approximately 1-2 sec with the expansion ratio of 20 – 26 times. Therefore, the foam with 60 phr of Fe_2O_3 has been chosen due to the highest amount of Fe_2O_3 loading, which means it could adsorb higher amount of H_2S.

In the scale – up section, the H_2S adsorption performance of the foam is tested to find the adsorption rate. The 60 phr of Fe_2O_3 foam is packed in the column with the bulk density of 0.015 g/cm^3, 0.015 g/cm^3 with the presence of water and 0.030 g/cm^3. In result shows that the behavior of the reaction is similar to the second – order reaction with the reaction rate constant (k) of 1.2×10^{-5} min^{-1}, 4.0×10^{-6} min^{-1} and 1.8×10^{-5} min^{-1} respectively. The k achieved from the experiment can be used to scale up the foam for the industrial use by the estimating equation: $1/C = 1/C_0 + kt$.

5.2 Suggestion

- Iso-butane gas must be concerned because of their high flammable and explosive properties.
- The polymer foam with more than 60 phr of Fe_2O_3 can be achieved by adjusting polymer foaming conditions.
- Adding some nucleating agent in order to easier occur of foam nucleation.
- Increase the surface area by cut the foam into small pieces to make it easier to absorb H_2S.

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APPENDIX A

Heat of reaction calculation

1. Heat of formation of the reactants is investigated as showed in Table A.1

Table A.1 Heat of formation and Gibbs free energy (25°C and 1atm)

Compound	ΔH_f^0(kJ mol^{-1})
Fe_2O_3 (s)	-824.2
Fe_3O_4(s)	-1118.4
H_2S (g)	-20.6
Fe_2S_3 (s)	-94.6
S (s)	0
H_2O (l)	-285.8

2. Find the reaction equation

$$H_2S\ (g) + \tfrac{1}{3} Fe_2O_3\ (s) \rightarrow \tfrac{1}{3} Fe_2S_3\ (s) + H_2O\ (l)$$

3. Find out if the reaction is endothermic or exothermic reaction by calculating the difference between heat of formation of the product and reactants.

$$\Delta H = \tfrac{1}{3}(Fe_2S_3) + (H_2O) - (H_2S) - \tfrac{1}{3}(Fe_2O_3)$$
$$\Delta H = \tfrac{1}{3}(-94.6) + (-285.8) - (-20.6) - \tfrac{1}{3}(-824.4)$$

ΔH= -22 kJ (at 25°C and 1 atm)

From the calculation the difference of heat of formation is -22 kJ which means the reaction is exothermic reaction.

APPENDIX B

Gibbs free energy calculation

1. Find the Gibbs free energy of chemicals in the system as shown in Table a.1

Table B.1 Gibbs free energy

Compound	ΔG_f^0(kJ mol^{-1})
Fe_2O_3 (s)	-742.2
Fe_3O_4(s)	-1015.5
H_2S (g)	-33.6
Fe_2S_3 (s)	-166.9
S (s)	0
H_2O (l)	-237.2

2. Find the reaction equation

$$H_2S\ (g) + \frac{1}{3} Fe_2O_3\ (s) \rightarrow \frac{1}{3} Fe_2S_3\ (s) + H_2O\ (l)$$

3. Find out if the reaction is spontaneous by calculating the difference between gibbs free energy of product and reactants. If the value is negative means the reaction is spontaneous, where the positive value means the reaction is unspontaneous.

$$\Delta G=\frac{1}{3}(Fe_2S_3)+(H_2O)-(H_2S)-\frac{1}{3}(Fe_2O_3)$$

$$\Delta G=\frac{1}{3}(-166.9)+(-237.2)-(-33.6)-\frac{1}{3}(-742.2)$$

$$\Delta H= -11.8\ kJ \qquad (at\ 25°C\ and\ 1\ atm)$$

From the calculation, the difference of gibbs free energy of the product and reactant is -11.8 kJ which means the reaction is spontaneous and can be occurred without the addition of external energy.

APPENDIX C

Batch foaming process

1. Set the temperature of heater for reaction while turn on the fan to make the uniform heating in the silicone oil bath.
2. Put the polymer sample into the reactor and close the reactor.

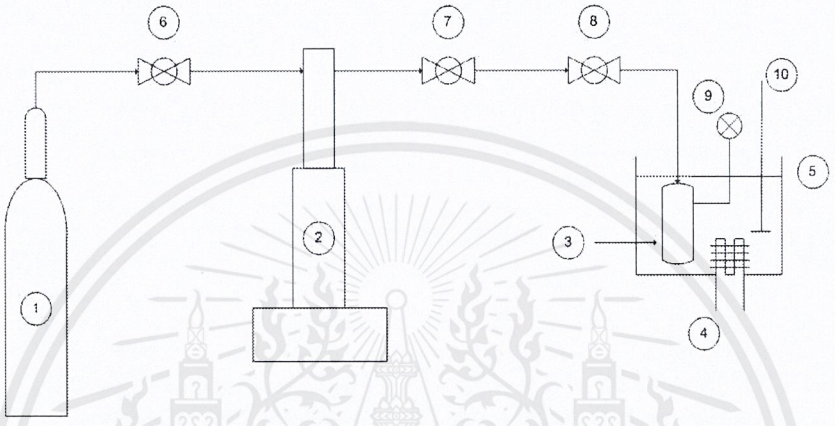

Figure C.1 Schematic of batch foaming process

(1) CO_2 gas
(2) reactor
(3) Silicone oil Bath
(4) Valve 2
(5) Pressure Gauge
(6) High pressure pump
(7) Heater
(6) Valve 1
(8) Valve 3
(10) Fan

3. Pump the carbon dioxide into the reactor with high pressure pump until the pressure reaches 1500 psi and leaves it for a while. Then eliminates the air out through the bottom of the reactor, which the final pressure should be higher than the atmospheric pressure to prevent the air backflow into the reactor.
4. Pump the carbon dioxide into the reactor again by using the pressure for foaming from the experiment. After achieved the target temperature of silicone bath, immerse the reactor into the silicone bath and leave it for a while.
5. Close valves 2 and 3 (no.7 and no.8 respectively), then take off the high-pressure reactor. Foaming process would occurs after reduce the pressure rapidly by open valve no.3. The pressure would decrease from the desired pressure to the atmospheric pressure in less than 5 seconds.
6. Take the polymer sample for further analyzed.

APPENDIX D

Density analysis by electronic density testing machine

The electronic density testing machine working based on the displacing of water by the object or the Archimedes' principle, which has the procedure described below.

1. Set the display into zero value (Set zero) as shown in Figure D.1
2. Weight the polymer foam at the top of the densimeter showed in Figure D.1 then press the button 'Memory' to record the value.
3. Weight the polymer foam in the water (the bubble on the sample surface should be eliminated). Press the button 'Memory' to calculate the density of the polymer foam.
4. The densimeter will show the density value in the unit of g/cm^3 (could be adjusted into the unit m^3 by pressing 'A' button).
5. Bring the polymer foam out of the densimeter.

Note: To take out the sample from the densimeter should be done by using the forceps, in order to prevent the water spill out and leads to the broken of the electronics equipment.

(1) Screen display (2) Densimeter

Figure D.1 Electronic Densimeter ; MD-200S

APPENDIX E

The foam morphology analysis by ImageJ

The foam morphology analysis procedure by imagej to analyze the thickness of foam cell wall is described by the following steps.

1. Open the imagej program, then select the SEM image by select 'file' and 'open' as showed in Figure E.1.

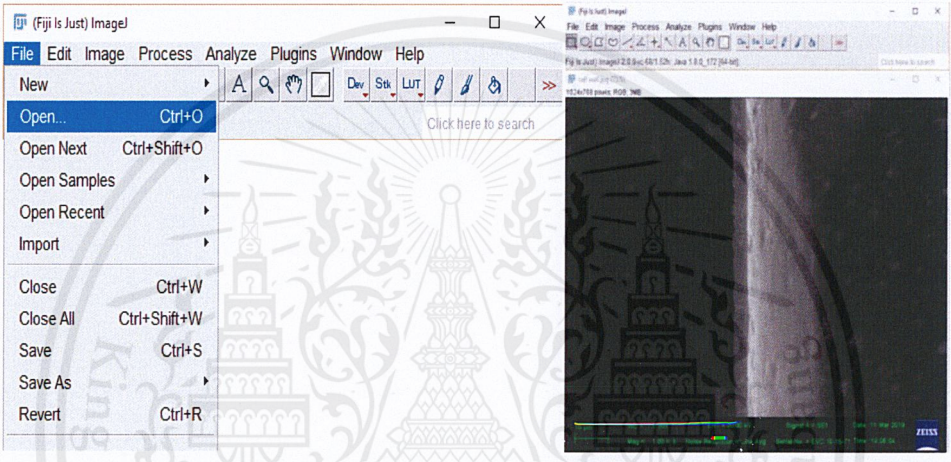

Figure E.1 Open the SEM image in the imagej program

2. Select 'Analyze' and 'Set scale' on the tool bar to adjust the scale as showed in Figure E.2

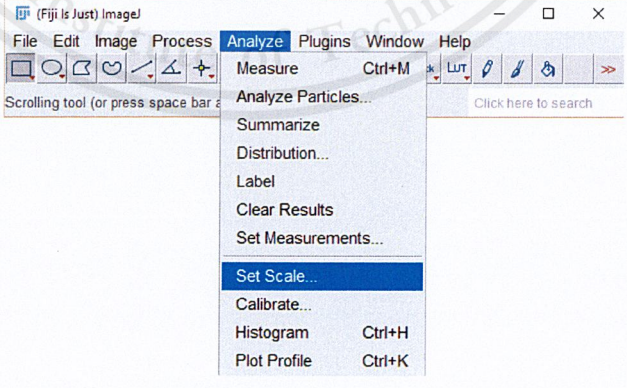

Figure E.2 Adjusting the scale

3. Fix the known distance, then key the known distance and select the unit in the ‘unit of length to fix the pixel of the image and the real distance.

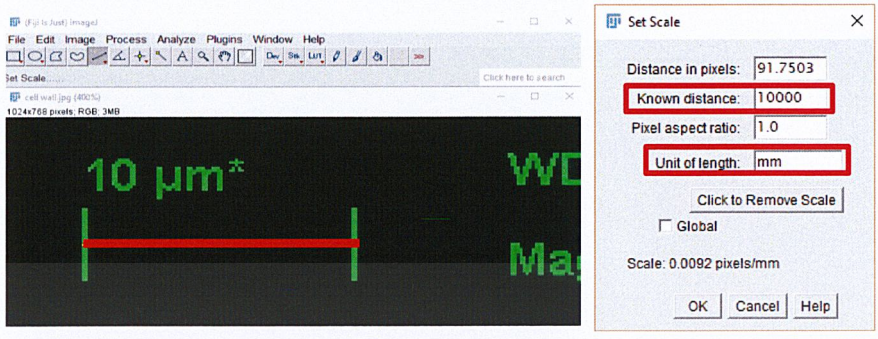

Figure E.3 Adjust the scale and unit

4. Draw the line that is equal to the thickness of the cell wall then going to ‘analyze’ and ‘measure’. The program will show the size of the line that we drew in the column ‘Length’ as shown in Figure E.4 According to the figure, the cell wall would have the thickness equals to 4,414.49 mm.

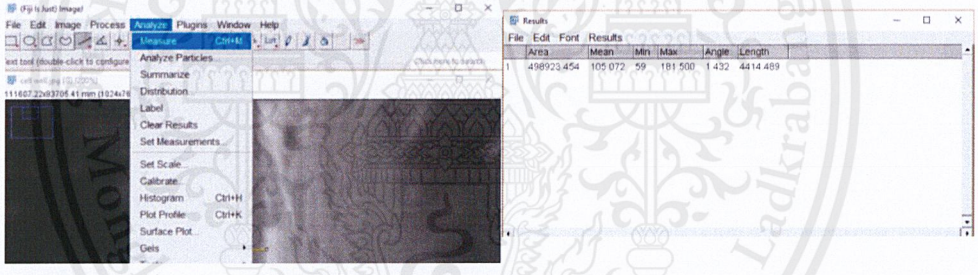

Figure E.4 Size measuring and length resulting

APPENDIX F

Estimate diffusion of H_2S in PE foam

Table F.1 Collision diameters and energy parameters for the Lennard-Jones equation

Molecule	Compound	$\sigma \times 10^{10}$ (m)	ε/k (K)
Cl_2	Chlorine	4.217	316.0
F_2	Fluorine	3.357	112.6
HBr	Hydrogen bromide	3.353	499.0
HCN	Hydrogen cyanide	3.630	569.1
HCl	Hydrogen chloride	3.339	344.7
HF	Hydrogen fluoride	3.148	330.0
HI	Hydrogen iodide	4.211	288.7
H_2	Hydrogen	2.827	59.7
H_2O	Water	2.641	809.1
H_2O_2	Hydrogen peroxide	4.196	289.3
H_2S	Hydrogen sulfide	3.623	301.1
Hg	Mercury	2.969	750.0
$HgBr_2$	Mercuric bromide	5.080	686.2
$HgCl_2$	Mercuric sulfide	4.550	750.0
HgI_2	Mercuric iodide	5.625	695.6
I_2	Iodine	5.160	474.2
NH_3	Ammonia	2.900	558.3

1. Calculation of diffusivity

$$10^{-3}\frac{E_D}{R_g}=\left(\frac{\sigma_{H_2S}}{\sigma_{N_2}}\right)^2\left[7.5\text{-}2.5\times10^{-4}\left(T_g\text{-}298\right)^{3/2}\right]\pm0.6$$

Where; $R_g = 8.314$ J/mol.K

$\sigma_{H_2S} = 36.23$ nm

$\sigma_{N_2} = 37.98$ nm

T_g of PE = 148 K

Find activation energy of diffusion (E_D)

$$10^{-3}\frac{E_D}{8.314}=\left(\frac{36.23}{37.98}\right)^2\left[7.5\text{-}2.5\times10^{-4}(148\text{-}298)^2\right]\pm0.6$$

$$E_D = 14.185\pm8.3 \text{ kJ/mol}$$

Find diffusivity of the corresponding completely amorphous material (D_0)

$$\log D_0 \cong \frac{E_D\times 10^{-3}}{R_g} - 5.0 \pm 0.8$$

$$\log D_0 \cong \frac{14.185\times 10^{-3}}{8.314} - 5.0 \pm 0.8$$

$$D_0 \cong 1.004\times 10^{-5} \text{ cm}^2/\text{s}$$

Estimate the diffusivity (D) of hydrogen sulfide in polyethylene (PE) at 298 K

$$D \cong D_0\exp(-E_D/R_gT)$$

$$D \cong 1.004\times10^{-5}\exp(-1706/298)$$

$$D \cong 3.264\times10^{-8} \text{ cm}^2/\text{s}$$

2. The time of adsorption in PE foam (assume adsorption in a slab)

Laboratory scale

$$t = \frac{b^2}{D}$$

Where; t = The time of adsorption
b = (The thickness of cell wall)/2
D = diffusivity

1. The time of adsorption (t) at condition (temperature of 120 °C and pressure of 1,500 psi)

$$b = 1.543 \times 10^{-4} \text{cm}$$
$$t = \frac{b^2}{D}$$
$$t = \frac{(1.543 \times 10^{-4})^2}{3.264 \times 10^{-8}}$$
$$t = 0.73 \text{ s}$$

2. The time of adsorption (t) at condition (temperature of 125 °C and pressure of 1,500 psi)

$$b = 1.414 \times 10^{-4} \text{cm}$$
$$t = \frac{b^2}{D}$$
$$t = \frac{(1.414 \times 10^{-4})^2}{3.264 \times 10^{-8}}$$
$$t = 0.61 \text{ s}$$

3. The time of adsorption (t) at condition (temperature of 130 °C and pressure of 1,500 psi)

$$b = 1.653 \times 10^{-3} \text{cm}$$
$$t = \frac{b^2}{D}$$
$$t = \frac{(1.653 \times 10^{-3})^2}{3.264 \times 10^{-8}}$$
$$t = 83.62 \text{ s}$$

Industrial scale

$$t = \frac{b^2}{D}$$

Where; t = The time of adsorption
b = (The thickness of cell wall)/2
D = diffusivity

1. The time of adsorption (t) at condition (mixing 60 phr of Fe_2O_3 temperature of 120 °C and pressure of 1,500 psi)

$$b = 1.741 \times 10^{-4} cm$$
$$t = \frac{b^2}{D}$$
$$t = \frac{(1.741 \times 10^{-4})^2}{3.264 \times 10^{-8}}$$
$$t = 0.928 \text{ s}$$

2. The time of adsorption (t) at condition (mixing 60 phr of Fe_2O_3 temperature of 120 °C and pressure of 1,500 psi)

$$b = 1.811 \times 10^{-4} cm$$
$$t = \frac{b^2}{D}$$
$$t = \frac{(1.811 \times 10^{-4})^2}{3.264 \times 10^{-8}}$$
$$t = 1.004 \text{ s}$$

3. The time of adsorption (t) at condition (mixing 60 phr of Fe_2O_3 temperature of 120 °C and pressure of 1,500 psi)

$$b = 2.207 \times 10^{-4} cm$$
$$t = \frac{b^2}{D}$$
$$t = \frac{(2.207 \times 10^{-4})^2}{3.264 \times 10^{-8}}$$
$$t = 1.49 \text{ s}$$

APPENDIX G

Details of gas data meter detector

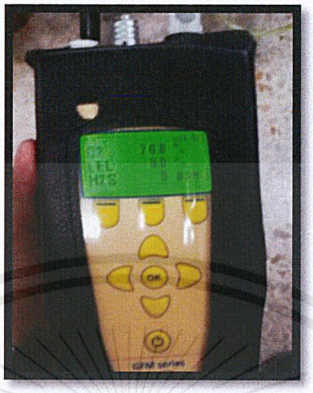

Figure G.1 Gas data meter detector (GFM435 Series)

Table G.1 Certification Ranges of gas measuring

Gas	Certification Ranges
CH_4	0 to 60%vol
CO_2	0 to 40%vol
O_2	0 to 20.9%vol
H_2S	0 to 5000 ppm
CO	0 to 2000 ppm
Flow	0 to 100 Lh^{-1}

Note:

- Borehole flow, borehole pressure and atmospheric pressure measurements
- Optional temperature and velocity measurement

Applications include:

- Laboratory analysis
- Biogas monitoring
- Compost monitoring
- Brewery monitoring

BIBLIOGRAPHY

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Academic Background: Bachelor Degree of Petrochemical Engineering

Working Experience:

1. Trainee (June – August 2018) : This internship program at Bangchak's refinery provides me knowledge in oil & gas field and also develop my soft skills. My assigned project during the internship program focuses on the reduction of energy consumption by reducing the amount of unnecessary unit operation. I have worked on the hydrogen sulfide removal unit where I have to study about the effects when the unit has been removed, the design of new process to overcome the effects, the equipment sizing and also the economic evaluation. During this project, I also learned the way to connect and work with other people. Moreover, in this internship program there were a lot of ice – breaking and CSR activities, also there was a social business project contest that trainees from different divisions have to work together with others to create the social business project which could solve socials problem in the financially sustainable way.

2. Collaborative Project with Ampelite (Thailand) Co., Ltd. at Yamagata University, Japan

3. Research team member (December 2017 - January 2018) : King Mongkut's Institute of Technology Ladkrabang collaborated with Ampelite (Thailand) Co., Ltd. to do a research project in the topic of "Degradation of Unsaturated Polyester Resins", which I have worked in this project as a part of the research team. I have to analyzed the samples at various conditions and design the problem-solving methods with the help from Yamagata University, Japan. This project also gave me an opportunity to train and do the research oversea with the supports from Ampelite (Thailand) Co., Ltd.

4. Plastic Injection research at Kyoto University, Japan Trainee (November 2018) : This short internship program at Kyoto University, Japan provides me knowledge in Plastic Injection. My assigned project during program focuses on injection and machine operation. I have to analyzed and designed the condition of machine to find the best condition.