Analysis Of Temperature Effect on Struvite Scales Controlling in A Vertical Reactor

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Abstract. Scale is forming and controlling in industrial applications causes the clogs or decreasing in volume flows in the pipes, so increasing maintenance cost can occur as a consequence. Struvite discovered as Magnesium Ammonium Phosphate Hexahydrate ($\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$) and referred as a scale due to the heating process of fluid through pipes. The aim of this research was investigated the temperature effect on process of scales forming and controlling. The process was used a vertical bulkhead reactor. The process starts from mixing the equimolar solutions of MgCl_2 , NH_4OH , H_3PO_4 with 1: 1: 1 of ratio concentration. Then, crystallization process was carried out with the condition at the variation of temperature (20-40°C), variation of pH value (pH 8-10) and variation of air inlet rate (0.25; 0.50; 0.75; 1.0; 1.25 L/min) while the feed rate was 25 ml/min. The process continues until the condition becomes steady state. The scale controlling process was determined by the presence of struvite deposits. The deposits were analyzed by using the XRF method to determine composition content. The optimal conditions were obtained at a temperature of 30°C, pH 9, and air rate of 1.25L/min.

Keywords: Temperature Effect, Struvite, Vertical Reactor

1 Introduction

Heat exchanger, the most important heat exchange equipment, was needed in the industrial applications such as heat-power industry, chemical, food processing, refineries oil and gas and other industries[9]. A blockage in the process piping occurs due to struvite scales which reduce the diameter of pipes, thereby impairing the flow of fluids, water, oil, gas, etc. These scales impair the pipes causing higher operational production cost; increasing temperature in pipes, higher pressure, increasing time of drainage and higher pipe maintenance cost [2,3]. Scales are the deposition of organics and inorganics compound mainly formed at the inner surface of heat exchange equipment which is through the crystallization process of mineral compound in the water [4]. Chemically-defined, scales formed due to changes of ionic composition, pH level, pressure, and temperature. For an illustration, some molecules will affiliate to form a crystal nucleus in a supersaturated solution. The formed crystal has lower ionic charges and tends to agglomerate to form scales [3,6]. Mineral ions dissolving process in water was affected by pressure, temperature, contact time between water and formation medium. Water has a limited ability to keep these ionic compounds in solution. At specific pressure and temperature conditions, when the solubility value was exceeded, the compound will not further

dissolve. However, the compound will separate from the solvent to form a solid [8].

Struvite or magnesium ammonium phosphate ($\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$) is a phosphate mineral compound. The struvite formation process starts with mixing Mg^{2+} , NH_4^+ , PO_4^{3-} ions in a solution. These reaction of ions in solution occur when the solubility product (K_{sp}) is lower than concentration of ions in solution.. Based on this [7], the reaction process to form struvite scales follows:

Several factors that affect the controlling of struvite scale ($\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$) process were pH, saturation level and presence of ions in solution. Based on [10], the ratio concentration of 1:1:1 for Mg:N:P ions in the liquid phase was achieved of 86.4% to form struvite deposits. Similarly, with that, the aeration column crystallizer was used to investigate the removal of PO_4 ions with ratio concentration of 1:1 (Mg:P) and obtained analysis result of 83.7% [1]. In an industrial application, struvite was found in pipelines as known as scales. The scales were indicated that contain magnesium ammonium phosphate compound. Industrial equipment that easily found scales were boilers or steam which are often flows with hot fluid.

This research was investigated to control the presence of struvite scales by using a vertical bulkhead reactor. The conditions of reactor were a volume of 498.75 mL, the

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height of 50 cm with an outside diameter (OD) of 5 cm and inside diameter (ID) of 2.5 cm. The reactor was equipped with bulkhead as an agitator. The mixing process was supported by the air that flowed into the reactor in counter-current flow with the incoming fluid flow. The function of these bulkheads was to break the air bubbles go into the reactor become smaller size bubbles, so that reaction between Magnesium Ammonium Phosphate Hexahydrate may occur in perfectly way. The results of struvite scales were analyzed by using the XRD method, which is analysis for the characterization of the material from the crystal. Meanwhile, the elemental composition of the crystals was analyzed by using the SEM method.

2 Research and Methods

With ratio concentration of 1: 1: 1, mixing solutions (MgCl_2 , NH_4OH , H_3PO_4) were prepared to control presence of struvite scales and the NaOH solution as a pH controller. The instrument of vertical reactors can be seen in figure 1 below :

Fig.1. Schematic Diagram of Vertical Bulkhead Reactor

The mixing solution was transferred to the reactor approximately $\frac{3}{4}$ of reactor's height. The air flow was fed into the reactor, as a counter-current flow, with variation of inlet air rate of 0.25 L/min; 0.50 L/min; 0.75 L/min; 1.0 L/min; 1.25 L/min. A NaOH solution was added gradually to reach the variation of pH value conditions (pH 8; 9; 10). The operation condition was maintained at variation temperature of 20°C; 30°C; 40°C. The process was continued until the condition become steady state. The XRF (*X-ray Fluorescence*) method was used to analyze the composition of struvite deposits.

3 Result and Discussion

XRF (*X-ray Fluorescence*) method was an element analysis method that forms material by interaction between X-Ray and compound. These 3 tables (Table 1-3) show the analysis result of struvite for different pH values (pH 8; 9; 10).

Table 1. Analysis Result of XRF method at pH value of 8

Temperature	Component (%)	Air Rate (L/min)				
		0.25	0.5	0.75	1.0	1.25
20°C	Mg	30.3	31.5	32.7	36.4	37.4
	P	22.4	23.6	26.8	27.6	28.5
30°C	Mg	33.5	30.2	32.3	34.4	38.9
	P	28.4	29.2	29.8	29.9	30.3
40°C	Mg	32.2	30.2	31.4	32.4	33.9
	P	27.8	28.2	28.8	29.4	29.7

Table 1, shows analysis result of mineral component at pH value of 8 with different temperature. The magnesium (Mg) content of 38.9% and phosphor (P) content of 30.3% was obtained at 30°C with air rate of 1.25 L/min.

Fig.2. Effect variation of temperature on percentage of Mg content (%) at pH 8

Figure 2, shows the correlation between temperature with the percentage of Mg content on struvite mineral. The high Mg content was obtained by using air rate of 1.25 L/min in different temperature. Similarly with Figure 2., on Figure 3. shows the correlation between temperature with the percentage of P content on struvite mineral. The high P content was obtained by using air rate of 1.25 L/min in different temperature. Depend on ^[11], air rate affects the formation and controlling process of struvite by using this bulkhead reactor. The purpose of the air rate was to increase the speed of mixing so reaction can occurs and lead to homogeneity solution. The higher of air rate, it will reaches a homogeneous condition perfectly. The highest percentage of Mg and P content was achieved at temperature of 30°C.

Fig. 3. Effect variation of temperature on percentage of P at pH 8

Table 2. Analysis Result of XRF method at pH value of 9

Temp.	Component (%)	Air Rate (L/mins)				
		0.25	0.5	0.75	1.0	1.25
20°C	Mg	36.5	36.8	38.4	39.6	40.2
	P	37.4	38.2	38.8	38.9	39.3
30 °C	Mg	40.5	40.2	41.4	42.4	43.9
	P	38.4	39.2	39.8	40.04	40.3
40 °C	Mg	35.5	36.2	41.4	42.4	41.9
	P	35.4	35.9	36.8	37.4	38.3

Table 2, shows analysis result of mineral component at pH value of 9. Magnesium content of 43.9% and Phosphor content of 40.3% were obtained with the operational condition at 30°C, pH 9 and air rate of 1.25 L/mins.

Table 3. Analysis Result of XRF method at pH value of 10

Temp.	Component (%)	Air Rate (L/mins)				
		0.25	0.5	0.75	1.0	1.25
20°C	Mg	28.5	28.8	29.07	29.4	30.9
	P	30.4	31.02	31.8	32.4	33.1
30 °C	Mg	30.5	30.2	32.4	32.4	33.9
	P	32.4	33.02	33.3	34.4	35.3
40 °C	Mg	28.5	29.02	29.4	30.04	31.07
	P	31.4	31.7	31.8	32.4	32.51

Table 3, shows analysis result of mineral component at pH value of 10. Magnesium content of 33.9% and Phosphor content of 35.3% were obtained with the operational condition at 30°C, pH 10 and air rate of 1.25 L/mins.

Fig. 4. Effect variation of temperature on percentage of Mg at pH 9

Fig.5. Effect variation of temperature on percentage of P at pH 9

The correlation between effect of temperature with air rate on struvite compound showed at Figure 4. and Figure 5. In Figure 4, the high component of Mg was obtained at temperature of 30°C with different air rate on 0.25; 0.5; 0.75; 1.0; 1.25 L/min. [5] shows that struvite solubility increase with the increasing of temperature until 35°C and then decreasing. The solubility product (K_{sp}) will increase from 0.436×10^{-4} to 5.920×10^{-14} at increasing of temperature 10 °C to 35°C. Depends on it, show that temperature has effect on the process of formation struvite. When the temperature solution of 35 °C then decreased to 10°C, struvite crystal will easily form in solution. In Figure 5. shows that the percentage of phosphor content decrease at temperature of 40 °C

Fig. 6. Effect variation of temperature on percentage of Mg at pH 10

Fig.7. Effect variation of temperature on percentage of P at pH 10

Figure 6 and Figure 7, shows the correlation between temperature with the percentage of Mg content on struvite mineral at different air rate. The high Mg content was obtained by using air rate of 1.25 L/min. Similarly with high Mg content, the high P content also obtains at air rate 1.25 L/min.

At variation of temperature, optimal temperature to control the struvite scale was obtained at 30°C. The pH value of 9 was obtained as optimal condition. The effect of pH value was one of the most important factors for the process to form the struvite deposits. When pH value was higher, more struvite deposits were forming. The optimal condition for this research was obtained at temperature of 30°C, pH value of 9, air rate of 1.25 L/min, analysis result for magnesium content of 43.9% and phosphor content of 40.3%.

4 Conclusion

The temperature, pH, air rate were very important factors that effect to control struvite scales on a bulkhead vertical reactor. The condition at 30 °C, pH 9, air rate of 1.25 L/min were determined as a optimal condition. On this optimal condition, the highest magnesium content of 43.9% and phosphor content of 40.3% was obtained as an analysis result.

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