# PINK GUAVA JUICE PASTEURISATION: FOULING DEPOSIT AND CLEANING STUDIES

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### **ABSTRACT**

Pasteurization is a common sterilization process in food industry to prolong the shelf life of the food product. However, fouling deposit problem is one of the major problems of the pasteurisation. This work provides fundamental studies on fouling deposition of pink guava juice (PGJ) and cleaning of it. A basic tubular heat exchanger set-up was used to perform pasteurization and cleaning. Pasteurisation was operated under industrial pasteurisation temperature of PGJ, which was at 93°C. Alkaline-based cleaning solution was utilised for ex-situ cleaning at different concentrations (0.2, 0.3, 0.4%v/v Na<sub>2</sub>O), 65°C and 1 l/min. Microstructure study of the deposits before and after cleaning was carried out using ESEM. Proximate analyses were performed to determine the composition of PGJ. A study on the hardness and stickiness of the fouling deposit was done using a texture analyser. The presence of seedstone in pink guava juice was analysed using a particle analyser. Cleaning performance was monitored via the changes of remaining deposit and clarity of the cleaning outlet. The findings shown that seedstone from pink guava juice ranging from 168 to 200µm and carbohydrate was found to be a major composition (47.7% of fouling deposit consists of carbohydrate). Comparison between the hardness and stickiness of the deposits at two different flow rates showed that fouling deposits were harder and denser at higher flow rate. Findings from this work provide basis knowledge for further study on fouling and cleaning of PGJ.

Keywords: Cleaning, Fouling Deposit, Heat Exchanger, Pink Guava Juice

# **1.0 INTRODUCTION**

Pasteurisation is a common sterilisation process in food industry to prolong the shelf life of the food product. However, not many people aware about the fouling deposit problem, which is one of the drawback of the pasteurisation. Pasteurisation, a process taken place at high temperature (>50°C) enables harmful bacteria to be killed and thus increase the shelf life and safety of the product. The temperature required to meet this is depending on the type of raw food materials that involved in the process. It is an essential step required in most food industries [1]. Heat exchanger is the common unit operation use for pasteurisation.

Most process of beverages and dairy products in food industries are operated for 24 hours a day and for almost every day. The heat exchanging mechanisms are taken place between the product and the heating medium. The primary objective of heating process is to kill the harmful bacteria. Even though the bacteria are being killed during this process, chemical reaction and the properties of the product changes as well. Very often, these changes produce scaling or precipitation on the inner surface of processing equipment which is known as fouling deposit.

Fouling deposit, a term use to describe the deposition of unwanted material formed on solid surfaces of processing equipment. Its formation can reduce the efficiency of the heat exchanger and consequently affects the pasteurisation process [2]. Fouling layer formed on the wall acts as a barrier to heat transfer. Besides that, it can reduce the pipe flow area, thereby resulting in pressure drop and inefficiency of heat transfer [2]. As a consequence, this will affect the industry's productivity and economy.

In this study, pink guava juice (PGJ) was used as the model fluid of the research. Pink guava is tropical fruit and Malaysia is

among the top in the world that producing pink guava puree for PGJ [11] and as ingredient for other food products. Furthermore, currently there is no publication on PGJ fouling deposit, according to an extensive literature survey by Tan (2009) [10] and Ho (2009) [4]. A basic tubular heat exchanger (THE) set-up for obtaining pink guava juice fouling deposit and for performing cleaning was constructed enabling the study to be conducted in continuous flow condition. The obtained deposit can be a physical model for fouling deposits from industrial scale heat exchanger. Fouling characteristics study was performed on the fouling deposits. The influences of different operating conditions on fouling formation were investigated while characterisation of the fouling layer such as texture, composition and microstructure were performed. Cleaning performance was also investigated under the influences of temperature, cleaning chemical concentration and flow rate. Alkaline-based detergent was used as alkaline is the best cleaning agent for carbohydrate-based and protein-based deposits [2]. The results from this study will provide basic knowledge on PGJ fouling deposit, where its formation rate and formulation for cleaning can be further understood.

# 2.0 MATERIALS AND METHODS

### Pink Guava Juice

Pink guava puree was supplied by Golden Hope Food & Beverages Sdn. Bhd., Sitiawan, Malaysia. It was diluted with distilled water at the ratio of 1:3:7 (sugar: pink guava: water), to produce pink guava juice. The percentage of Brix used was in the range of 7.0-11.0 and the Brix set for this experimental study was 11° Brix and the Brix percentage of the pink guava juice was determined using the refractometer.

# **Cleaning Chemical Solution**

A single stage cleaner, Principal (ECOLAB) was used to remove fouling deposit. It is an alkaline-based detergent that is formulated for the cleaning of food, dairy and beverage processing equipment. The properties of Principal are shown in Table 1. The main cleaning agent in Principal is sodium oxide (Na<sub>2</sub>O). Principal was diluted with distilled water to obtain three chemical concentrations (0.2%v/v, 0.3%v/v, 0.4%v/vNa<sub>2</sub>O).

### Basic Tubular Heat Exchanger (THE) Set-Up

A rig constructed of 304 stainless steel pipe with the outer diameter of 22mm and it was joined by Type A/ non manipulative, British standard compression fittings. These fittings were utilised in developing the rig that resembles a concentric tube exchanger.

The design of this heat exchanger (Figure 1) was based on the concept that the heat from the heating medium will transfer heat to the stainless steel pipe and eventually to the pink guava juice in the pipe in order to pasteurise the pink guava juice to the desired temperature. The pink guava juice was prepared and stored in a tank (20L) before being pumped into the tube that is immersed in glycerol. The heat exchanger contains 3 passes with length of 0.17m for each pass to provide a sufficient holding time for heating. One K-type thermocouple was placed near the outlet of the heat exchanger and another was placed in the heating medium to monitor the temperature of the pink guava juice and heating medium, respectively. Pink guava juice is then recycled to the tank.

Property	Description
Color	Light yellow
Specific gravity at 68(20)	1.237
Pounds per gallon	10.20(4.67kg)
рН	
1.0% solution	12.3
0.1% solution	11.1
Alkalinity	
Active as $Na_2O$	12.2%
Total Na <sub>2</sub> O	14.0%
Chlorine	3.0%

 Table 1: The properties of principal

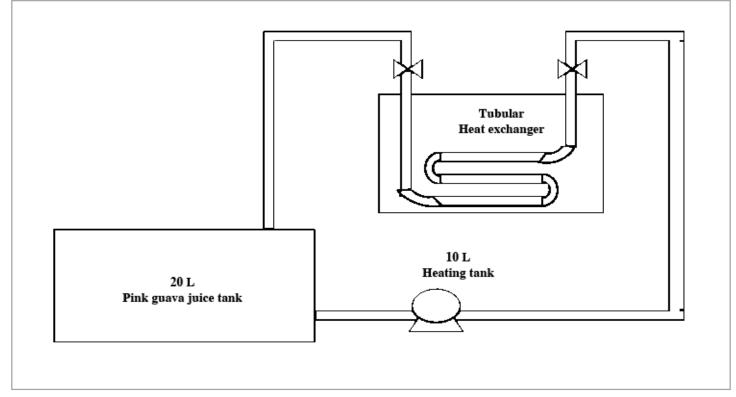


Figure 1: Basic tubular heat exchanger (THE) set-up

During cleaning of the THE, the tank was filled with cleaning chemical solution and the temperature of the heating tank was set to the desired cleaning temperature.

#### Temperature and Time for Pasteurisation

Pink guava juice is pasteurised at temperature range of 93°C-97°C and with holding time for 30s, according to the normal industrial practice. This information was obtained from Golden Hope Food & Beverages Sdn. Bhd and it provides a benchmark for the lab scale study. While the flow rate and pasteurisation period were based on the experimental capacity, which were 0.5 and 1 liter/min for the flow rate and the pasteurisation period was set for 1 hour.

#### **Cleaning Methods and Parameters**

Here, two methods were used to perform cleaning: (a) exsitu method with a shakable water bath [5] and (b) in-situ method with a basic tubular heat exchanger [10]. The first method was employed to perform preliminary cleaning studies in which then an optimum cleaning chemical concentration can be found. Then, the optimum cleaning chemical concentration was applied to the second method. Cleaning was started with the first method because the second method requires more cleaning solution, thus this procedure can reduce the experimental number for the second method and the cleaning chemical usage.

For the first method the cleaning parameters were Principal concentrations  $(0.2\% v/v, 0.3\% v/v, 0.4\% v/v Na_2O)$  and 50 rpm of shaker speed (to simulate the flow at 1 l/min [5]). While

for the second method, the cleaning parameters were 0.4% v/v Na<sub>2</sub>O (optimum concentration) and 1 l/min of cleaning solution flow rate. The temperature applied for both methods was 65°C. Principal supplier has recommended the cleaning temperature is between 60°C and 66°C for obtaining an optimum cleaning performance.

#### **Proximate Analysis**

This analysis was carried out to determine the compositions of moisture, fat, ash, fiber, protein and carbohydrate contents. Moisture content was determined by using the oven method. Fat content was determined by using the Soxhlet method, while protein content was examined using the Kjeldahl method. Determination of ash was measured by drying the sample in a muffle furnace up to 550°C. The fiber content analysis was carried out according to the procedures described by AOAC, 1995 [3].

#### **Texture Analysis**

For texture analysis, the hardness and stickiness of pink guava juice fouling deposit were examined by using the texture analyser (TA-XT Plus, Stable Micro Systems, UK). The parameters and specification of texture analyser for performing hardness and stickiness analyses were stated in Table 2.

#### Particle Analysis

The presence of seed-stone in pink guava juice was analysed for the size distribution. Pink guava puree was sieved with cheesecloth to separate the seedstone from the puree. The sieved residue was then laid flat and distributed evenly on a thin layer of aluminium foil before drying in the oven at 40°C for 24 hours. The dried residue was scrapped with spatula and kept in petri dish. The sample was stored in the desiccators prior to particle analysis. In analysing the particle size of the seedstone, particle analyser (Scirocco 2000, Malvern, UK) was used.

### **Cleaning Monitoring Techniques**

Cleaning progress was quantified: (*a*) by weighing the fouling deposit before and after the cleaning and (*b*) by measuring the clarity of the liquid outlet. Electronic weighing machine (Cartorius BS224S) was used for the first technique. The clarity of the liquid outlet was measured in relation to the light absorbance level. This technique was based on Beer-Lambert law that the light absorption of solution is directly proportional to its concentration. Thus, the greater the concentration of the remaining dissolved fouling deposit in the cleaning solution outlet, the greater the amount of light is absorbed. UV-Vis Spectrophotometer (UV mini 1240) was utilised to measure the light absorbance level.

#### Microstructure Analysis

Critical point drying was chosen as the choice of fixation technique for pink guava juice fouling deposit. Standard critical point drying (CPD) protocol was used for preparing the samples. After fixation with 4% glutaraldehyde in 0.1 M cacodylate and 2% buffered osmium tetroxide, the samples were dehydrated through a graded series of acetone (30%, 50%, 75% and 95% - once for 10min at each step), and then were immersed in 100% acetone for three times at 15 min each. The samples were then transferred to Bal-Tec CPD 030 critical point dryer (BAL-TEC AG, Switzerland) using liquefied carbon dioxide as transitional fluid before coated with gold by a sputter coater (Bal-Tec SCD 005, BAL-TEC AG, Switzerland). Then the dried sample was transferred to ESEM (XL30 Environmental Scanning Electron Microscopy, Philips, Holland) chamber, to obtain the ESEM micrograph for microstructure analysis.

# 3.0 RESULTS AND DISCUSSIONS

Fouling deposits from basic THE set-up differ physically from those obtained from shakable water bath system [4]. Figure 2 shows the fouled heat exchanger tubes, which were obtained at different flow rate conditions. For fouling deposit study in the basic THE, the controlled parameter was the flow rate. The flow rates used were 0.5 liter/min and 1 liter/min, which were the same flow rates used in the shakable water bath system (the flow rate is equivalent to the shaker speed) [5].

From Figure 2, the amount of fouling deposit formed was higher at the flow rate of 1 liter/min compared to the fouling deposit formed at 0.5 liter/min. The area of deposition was mainly at the

 Table 2: Parameters and specification of texture analyser for hardness and stickiness analysis of pink guava juice fouling deposit

Parameter	Value	Description
Probe	P20	The size of the probe used: cylindrical; 20mm
Test Mode	Compression	Initial probe direction and force polarity
Pre-test speed (mm/s)	1	Probe speed while searching for trigger point
Test-speed (mm/s)	2	Speed of approach to target distance after triggering
Post-test speed (mm/s)	2	Speed at which the probe returns to start point
Target mode	Strain	Target parameter
Strain (%)	30	Specify target strain base on trigger height
Trigger type	Auto	The initiation of data is defined
Trigger force (g)	5	Amount of force for TA to initiate data capture
Trigger distance (mm)	2	Pre-travel distance before initiating data capture

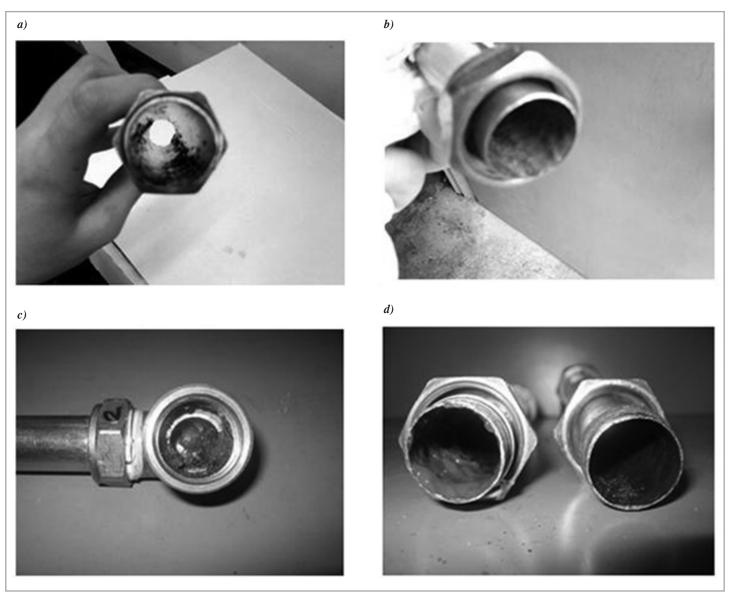


Figure 2: PGJ fouling deposit after heating process at flow rate of: (a)-(b) 0.5 liter/min and (c)-(d) 1.0 liter/min

Component (%)	Pink Guava Juice	Fouling Deposit
Moisture Content	89.09	50.3
Ash	0.54	0.83
Protein	0.93	1.22
Fiber	0.73	1.3
Fat	0.2	0.09
Carbohydrate	8.51	46.27

Table 3: Compositions of PGJ and its fouling deposit

	Hardness, Force (g)		
Test	Fouling Deposit, 0.5 liter/min	Fouling Deposit, 1 liter/min	
1	23.9021	70.8240	
2	30.3161	71.3198	
3	28.8171	68.3017	
4	27.6751	69.8121	
5	29.5616	70.2205	
6	26.7268	69.1772	
Mean	27.8331	69.9426	
Standard Deviation	2.3162	1.0997	

# Table 4: Hardness of PGJ fouling deposit

# Table 5: Stickiness of PGJ fouling deposit

	Stickiness, Force (g)		
Test	Fouling Deposit, 0.5 liter/min	Fouling Deposit, 1 liter/min	
1	15.2568	32.3685	
2	20.8214	33.5813	
3	20.108	33.9749	
4	20.4647	34.4659	
5	18.0391	34.9702	
6	19.0736	35.5021	
Mean	18.9606	34.1438	
Standard Deviation	2.0795	1.1071	

lower tube, which was the result of sedimentation and non-fill tube. The amount of deposition through the tube was not uniform. The top deposit texture was soft and soggy, resembled semi-solid. While the lower deposit texture was hard and quite dry. Generally different textures of the deposit are influenced by the temperature and the duration of heating, which then promotes ageing [6]. The fouling deposit from this experimental set-up is more appropriate to represent the fouling deposit from industry than the deposit from shakable water bath system. This is due to the experimental condition that includes the effect of continuous flow state.

Table 3 shows the results of the proximate analysis of PGJ and PGJ fouling deposit. From the table, the solid content of PGJ is approximately 10.92%. The solid content consists primarily of carbohydrate, which is approximately 77.94% of solid content. While the major composition of PGJ fouling deposit is carbohydrate (46.27%). Thus PGJ fouling deposit is carbohydrate-based deposit. Grassoff (1997) [7] classified fouling deposit based upon its main composition and he has concluded that

carbohydrate-based deposit is easy to remove but if cameralisation occurred, the removal process will be harder. Other researches on carbohydrate-based fouling deposit were using tomato paste [8, 9] as their material of study.

Tables 4 and 5 show the hardness and stickiness of PGJ fouling deposits that were obtained from the basic THE set-up. The hardness of the fouling deposit from heating process at 1 liter/min is 251% higher than the fouling deposit from the heating process at 0.5 liter/min. Thus, the velocity of the flow has a major effect on the hardness of the fouling deposit. A faster flow rate resulted in a harder fouling layer. According to [2], a biofilm/ fouling layer can be more compact and dense under high velocities. At low velocity, the structure can be more open and fluffy, resulting in fouling deposit structure that was not dense and hard. The open structure enables more air to be trapped within the structures. Similar trend was also observed for stickiness, in which fouling deposit formed at 1 liter/min was stickier than the deposit formed at flow rate of 0.5 liter/ min (Table 5).

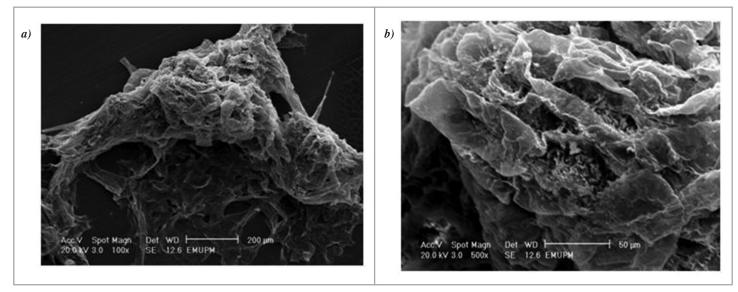


Figure 3: ESEM micrograph of pink guava juice at: a) 100x and b) 500x magnification

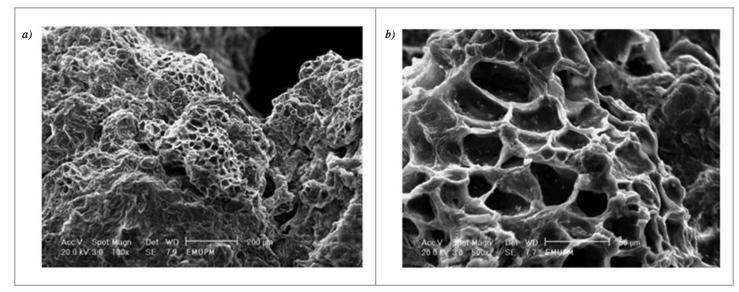


Figure 4: ESEM micrograph of PGJ fouling deposit at: a) 100x and b) 500x magnification

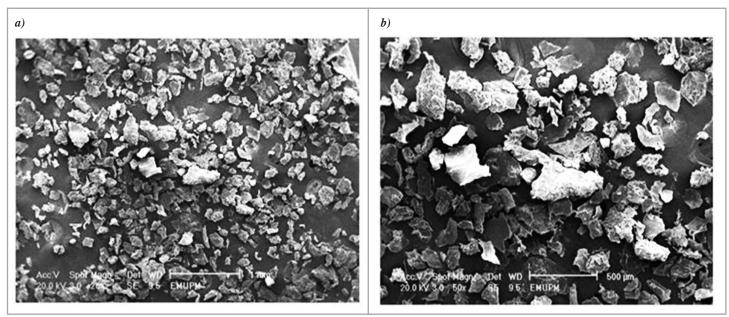


Figure 5: ESEM micrograph of the particles, 'seed-stone' at: a) 25x and b) 50x magnification

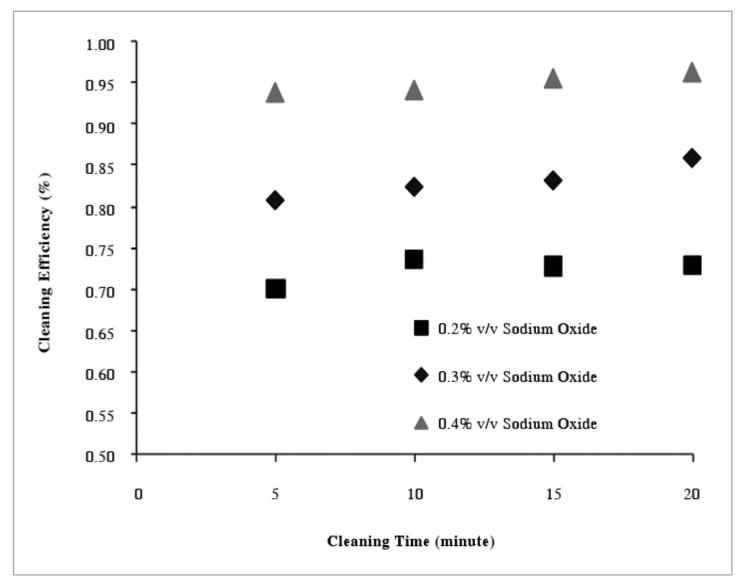


Figure 6: Cleaning efficiency for shakable water bath cleaning method (ex-situ method) at 65°C, 50 rpm and different cleaning solution concentrations

Cleaning time (minute)	5	10	15	20
Before cleaning				
After cleaning with 0.2%v/v Na <sub>2</sub> O				
Before cleaning				
After cleaning with 0.3%v/v Na <sub>2</sub> O				
Before cleaning				
After cleaning with 0.4%v/v Na <sub>2</sub> O				

 Table 6: Condition of fouled plates from ex-situ method before and after cleaning at 65°C, 50 rpm and at different cleaning solution concentrations

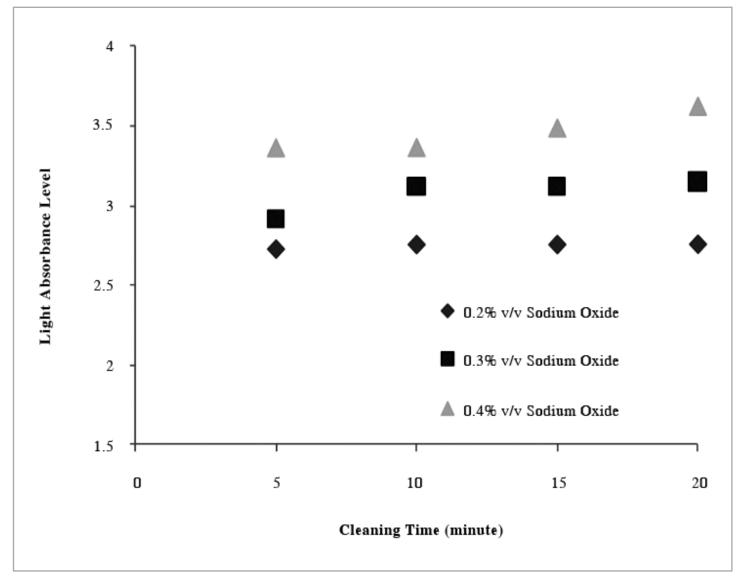


Figure 7: Light absorbance level for the outlet of shakable water bath cleaning method (ex-situ method) at  $65^{\circ}$ C, 50 rpm and different cleaning solution concentrations

The microstructure of PGJ and PGJ fouling deposit are dissimilar. Figure 3 shows the ESEM micrograph for PGJ. The micrograph shows that PGJ contains very fine sacs of carbohydrates and networks of fibers. Seed-stones were not observed in the micrograph (Most probably they were covered by the sacs and network of fibers). There was a distinct characteristic between the juice and the fouling deposit. In the fouling deposit as shown in Figure 4, the sacs were no longer observed. A dough like structure was observed. Besides, the fouling deposit looked dense, hard and porous to some extent. The observation may lead to the conclusion that heat treatment of pink guava juice has led to the denaturation of some of the organic matters, which is mostly carbohydrates as shown by the proximate analysis results in Table 3. The sacs of carbohydrate burst due to retrodegradation and form aggregates which settle with the seed-stones that were entrapped in the network. The microstructure of fouling deposit from this experimental set-up was developed gradually due to the continuous flow and thermal effect, which differ from those deposits obtained from the shakable water bath system [5]. Thus the microstructure is comparable to other fouling deposit obtained from similar flow system, such as milk fouling deposit [8].

The uniqueness of PGJ fouling deposit is due to the presence of seed-stone, which is insoluble in water. The sedimentation of the seed-stone will influence the hardness of the fouling deposit. This characteristic is not present in well-defined milk fouling deposit. Figure 5 shows the micrograph for the seed-stone. The size of the seed-stone is ranging from 168 to 200µm.

When discrete and fine particles like seed-stones are present in a fluid, they cannot take part in any deformation that the fluid may undergo. The result is an increasing resistance to shear, which is greater than a pure fluid [2]. Therefore the suspended solid resulted in an increased resistance to shear and hence suspended on the wall of the heat exchanger in which fouling deposit was eventually formed.

Carbohydrate-based fouling deposit can be cleaned by water alone but the cleaning performance is not efficient [8, 9]. Cleaning of PGJ fouling deposit with water alone requires high temperature and shear stress effect to increase the removal activities, but only

Cleaning time	Pipe Sections         Pipe Sections		
(minute)	Pipe joints	Cylinder tubes	
0			
10			
15			
20			

Table 7: Condition at different pipe sections after cleaning at 65°C, 0.4%v/v Na2O and 1 l/min

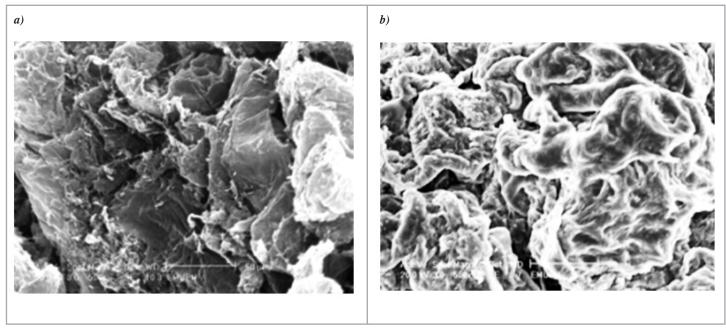


Figure 8: ESEM micrograph of PGJ fouling deposit: a) after exposed to 0.2% v/v Na2O and b) after exposed to 0.4% v/v Na2O for 20 minutes at 65°C (500x magnification)

50% removal was observed after cleaning for one hour [10]. In this study, cleaning was performed with formulated cleaning solution, Principal. From the preliminary cleaning study with shakable water bath cleaning method, cleaning performance was greatly improved when the cleaning solution was applied. Table 6 shows the changes on the fouled plate before and after cleaning at 0.2, 0.3 and 0.4% v/v Na<sub>2</sub>O. The pictures illustrate that less than 20 minutes is required to remove almost all of the deposit. This indicates that the chemical solution reacted better than water as cleaning agent. The chemical reaction weakens the intermolecular forces. The deposits remained on the plate is reduced when the chemical concentration increases from 0.2% v/v to 0.4% v/vNa<sub>2</sub>O. This suggests that higher concentration provides both a greater driving force for chemical diffusion and a higher reaction rate.

Cleaning efficiency was determined by weighing the fouling deposit before and after being exposed to the cleaning agent. The cleaning efficiency increases 10% for every increment in chemical concentration. Figure 7 illustrates that when more deposits are detached, the greater the concentration of remaining solution and the greater the amount of light is absorbed. Figure 6 and 7 show that the ex-situ cleaning with 0.4% v/v Na<sub>2</sub>O has the best cleaning efficiency and highest light absorbance level. Thus cleaning study with in-situ method was only performed at this concentration.

Table 7 clearly illustrates the remaining fouling deposit at different sections of pipe tubes and joints after cleaning for 10, 15 and 20 minutes. Less deposit was found with the increase of cleaning time. Longer duration of cleaning with  $0.4\% v/v Na_2O$  results in better cleaning effect.

Figure 8 indicates that chemical concentration affecting the deposit microstructure. Figure 8 shows that the deposit dissolved

away and formed amorphous clumps after being contacted with cleaning chemical. The morphology changes of the deposits vary with detergent concentration. Increase in chemical concentration produces more void with bigger size. Figure 8(b) illustrates that there is a dramatic change in the deposit structure after exposed to  $0.4\% v/v Na_2O$ . This observation demonstrates that increase in chemical concentration might reduce the strength of the deposit structure, thus assisting the cleaning process.

### **4.0 CONCLUSIONS**

This study used PGJ as a model fluid to obtain PGJ fouling deposit, which has not been studied previously. PGJ fouling deposit is classified as carbohydrate-based deposit. PGJ fouling deposit is unique compared to milk fouling deposit, due to it is containing particles, which are known as seed-stone. These fine particles (168 to 200µm) were embedded in the fouling deposit and were observed only on the fouling deposit, as heating process denatured some of the components that covering the seed-stone. The presence of the seed-stone might influence the hardness of the fouling deposit. A basic THE set-up was developed to ensure the deposit is similar to industrial-based deposit. The texture of the fouling deposit at different flow rates revealed that fouling deposit formed during a faster flow rate process has a harder and stickier texture compared to fouling deposit formed during a slower flow rate due to the fouling layer may be more compact and dense under high velocities. While in the presence of low velocity, the structure may be more open and fluffy. The cleaning process was only performed at flow rate 1 l/min (50 rpm) for shakable water bath method (ex-situ method) and 1 l/min for the basic THE set-up, as both methods used the preliminary designed rigs that can be improved in the future. Cleaning results

show that chemical-induced cleaning is better than cleaning with water alone, in term of cleaning time and cleaning efficiency. Chemical-induced cleaning can also reduce the water and energy consumption in cleaning. From both ex-situ and in-situ methods, cleaning was significantly improved when the cleaning chemical concentration was increased. This can be explained by the action of the cleaning agent in weakening the microstructure of the fouling deposit, hence assisting the cleaning. In conclusion, this work has discovered some important characteristics of PGJ fouling deposit and the basic THE set-up has successfully produced the PGJ fouling deposit. Thus findings from this work can benefit PGJ industry in Malaysia. Findings from this work will provide the basis for future study on properties of PGJ fouling deposit and its cleaning aspects.

# **5.0 ACKNOWLEDGMENT**

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