THE INFLUENCE OF FROTHER IN FLOTATION USING ELECTROLYSIS FOR BATIK DYEING WASTE SEPARATION

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Abstract

Batik waste can increase water characteristics such as turbidity, color and Total Suspended Solid (TSS). A technique for separating Batik from the liquid so that its turbidity, color and TSS decrease is needed. Flotation studies have been conducted using electrolysis to produce the bubbles to separate waste synthetic dye staining result of Batik from the bulk liquid. Research carried out by electrolysis with 316L stainless steel electrodes, inside an acrylic pipe with a height of 100 cm, and 8,4 cm in diameter with a voltage 15 V. Solid aluminum sulfate as a reagent was added to coagulate Batik waste as much as 1 gram per 10 ml of Batik waste. Batik waste was mixed with distilled water beforehand. Frother used was pure ethanol as much as 0.1% v/v. From the research it was discovered that flotation of Batik waste can be used for Batik waste separation with the addition of alum. Alum was proved capable of acting as collector in this type of waste separation. Ethanol as frother used was proved capable of making stable froth and increases the separation efficiency.

Keywords: bubble dynamics, Batik waste, flotation, electrolysis, electroflotation

1. Introduction

Batik is one of Indonesian cultural heritages. It has been admitted by UNESCO. Batik demand in clothing has been increasing the waste generated. Batik waste can degrade water quality by increasing its turbidity, color and Total Suspended Solid (TSS). Several methods have been used by Indonesian researchers to solve this problem such as coagulation, sedimentation, adsorption and filtration. The method is usually used in combination, e. g. by Setyaningsih (1995) who combined the method of coagulation, sedimentation and adsorption (Setyaningsih, 1995). Most often method used was adsorption, as has been done by Darmawanti and Rahmawati (2009) and Basuki (2011). Another method that has been used is electrolysis by Riyanto (2011). Actually, Setyaningsih (1995) conducted several methods, including flotation. She found that the method of coagulation-sedimentation-adsorption were more effective than the coagulation-flotation. Nevertheless, review of flotation parameters is not much discussed inside the flotation method used. As a result, the cause of the effectiveness is hardly investigated. The aim of this study is to investigate the bubble characteristics that influence the result of flotation of the Batik waste.

Bubble flotation efficiency is affected by three parameters, i. e. the probability of collision between bubbles with particles, the particles stick to the surface of the bubbles and the particles carried by bubbles (Matis, 1995). The higher the probability value, the higher the efficiency of the flotation. Smaller bubbles with higher numbers can increase the surface area so that the probability of collision between particles and bubbles can be improved. In addition, the terminal velocity of smaller bubbles will be able to increase the probability of collision and attachment of particles to bubbles.

2. Methodology

In this study there were two kinds of data taken, i. e. the characteristics of the Batik waste liquid before and after flotation (turbidity, color and TSS) and the bubble characteristics (diameter and velocity).

2.1 Apparatus

Set up equipment for fluid characteristics is shown in Figure 1, and for the bubble characteristics is shown in Figure 2.

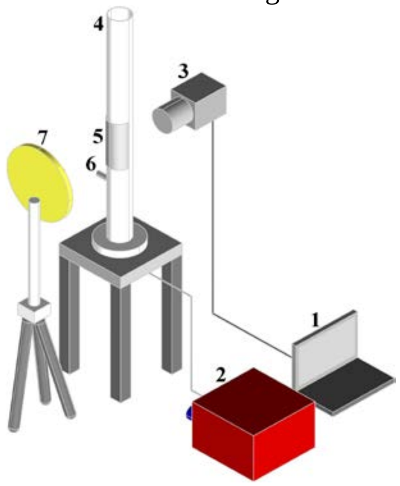

Figure 6. Experimental set up (1-computer; 2-DC Power Supply; 3-camera; 4-acrylic pipe; 5-diffusive screen; 6-faucet; 7-background light)

Acrylic pipe used had a length of 100 cm with inside diameter of 8.4 cm and thickness of 0.6 cm. At about 25 cm from the bottom of the pipe, the pipe was drilled and faucet was mounted with hose connected for sampling.

Bubbles were generated by electrolysis. Thus, they were hydrogen and oxygen bubbles. Electrode used was 316L stainless steel. Selection of 316L stainless steel was due to its resistance to wear in the electrolysis. Electrodes consisted of 5 plates that had the same width and thickness, which were 5 cm and 0.1 cm. Mean while it had different length, i. e. 2 pieces of 5.4 cm, 2 pieces of 6.9 cm, and 1 piece of 8.2 cm in increments. DC Power Supply was KPS3030DA of ATTEN instrument.

The design was adapted to the size and profile of the cross section of the pipe acrylic used for flotation of Batik waste, which was round. The plates were mounted parallel to the pipe on a styrofoam to maintain the position and distance interplate. Interplate distance was about 1.5 cm. The middle (longest plate) and edge (the two shortest plate) were the cathode with a total area of about 116 cm^2. While the rest was the anode plate with a total area of approximately 98 cm^2. The dimension of the cathode was 136 cm^2, but since most of the area was covered by styrofoam, it was reduced to 116 cm^2, as well as the anode.

The camera was a Nikon D5000 with AF-S lenses Nikorr 18-55mm f3.5-5.5G VR. Bubbles generated by electrolysis would be very small and micro-sized (except bubbles that have undergone merger with another bubble). Lens that was fitted normally will make it hard to see the movement of bubbles. To overcome this, a technique called macrophotography was used. It is a technique in using an extension tube to have a magnification required to see the bubbles. Extension tubes (ET) were mounted between the lens and the camera. Set up camera with ET is shown in Figure 2. Fluid and the form of cylindrical pipes can cause optical distortions. So, to calibrate the distance bubble had through, ruler was inserted into the liquid. Figure of the ruler which was put into the liquid can be seen in Figure 3.

Figure 7. Camera, lens and manual ET

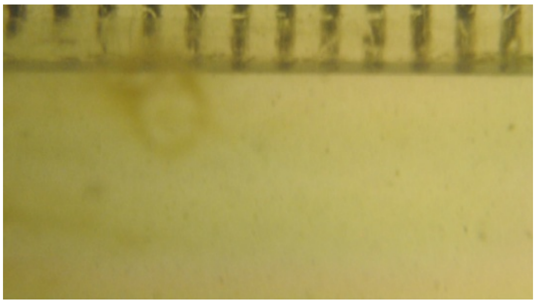

Figure 8. Ruler inserted into the liquid mixture (horizontal view)

Figure 3 is an example of an image taken with a camera and lens plus extension tube. It has a resolution of 1280 x 720 pixel. The distance between the lines on the ruler in Figure 3 is 1 mm. From this example, it can be said that camera set up acts like a microscope.

Back lighting technique was used by placing diffusive screen on the pipe on the opposite side of the mounted camera. Lights used for velocity bubbles data collection were two lamps of 18 W. Diffusive screen was taped to the back of the acrylic pipe in a certain area of the pipe to be recorded. This was done in order to spread the light throughout the area.

High shutter speed camera makes the camera lack of light, so 1500W halogen lamps without attaching diffusive screen behind the acrylic pipe area was used. If the diffusive screen was attached, there was only a black image on the camera's LCD. The use of this high-powered halogen lamps generated heat. So to overcome this, a transparent glass has been placed between acrylic and light pipes. To keep the temperature did not rise, a fan was mounted to flow the air to the area of the acrylic pipe near the lamp.

2.2 Materials

Acrylic pipe was filled with the mixture of 500 ml Batik staining waste (produced by Batik production center, D.I. Yogyakarta, Indonesia), 3500 ml of distilled water, 50 grams of alum and with or without addition of pure ethanol (pro analysis) as much as 0.1% v/v. The addition of ethanol as much as 0.1% v/v is based on study conducted by Shakir et. al. (2010) for reduction of Rhodamine B and Thoron using electrolysis. Shakir et. al. (2010) found that the percent reduction of Rhodamine B and Thoron drastically incresed from 75% to 99.5% and 99.9% for Rhodamine B and Thoron, respectively.

2.3 Experimental procedure

The liquid mixture was poured into the pipe as much as 150 ml. Experiments were performed with a voltage variation of 15 V for two types of liquid mixture, i. e. the mixture of Batik waste, alum and distilled water without ethanol and with ethanol. Samples were taken to the Health and Environment Laboratory, Department of Civil Engineering, Universitas Indonesia, to examine its turbidity, color and TSS.

From some measurements it was known that the pH of the liquid after flotation was not much different from before flotation, which was about 5. This value was effective based on flotation that was carried out by Hanotu et. al. (2012) for the separation of algae. The bubble characteristics (diameter and velocity) data collection was done after flotation of Batik waste. Thus, the liquid that was used for data collection of bubble velocity and diameter was the liquid of the flotation results. The data was processed using ImageJ software for the picture files and using Virtual Dub for the video files.

3. Results and Discussion

Figure 4 shows a sample Batik waste that has been mixed with alum before flotation. Based on Figure 4, it could be seen that the batik waste particles combine into particles with a larger size. This is due to the addition of alum which acts as a coagulant/flocculant.

Figure 9. The liquid mixture after poured into the acrylic pipe and allowed to stand for 5 minutes

Figure 5 shows the samples results of flotation using the electrolysis for 2, 4, 6, 8, 10 and 12 minutes with voltage of 15 V liquid without ethanol. In Figure 6, from left to right are the sample liquid before adding ethanol, after adding ethanol, and after electrolysis run for 2, 4, 6, 8, 10, and 12 minutes, respectively, for liquid with ethanol.

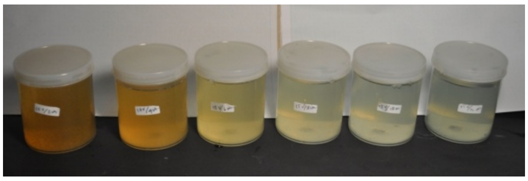

Figure 10. Samples of flotation with 15 V voltage

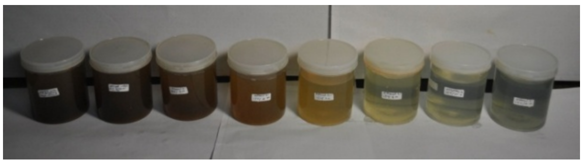

Figure 11. Samples of flotation with 15 V voltage, liquid plus ethanol

The value of the turbidity, color and TSS reduction For the 15 V voltage were 63.8%, 78.56%, and 95.98%, respectively. or samples with the addition of ethanol voltage of 15 V, the percent reduction of turbidity, color and TSS, i.e. 90.26%, 89.6% and 97.16%, respectively. This shows the influence of the addition of ethanol. From the experiments conducted, froth formed in this experiment was more stable than the froth without ethanol. In the experiment without the use of ethanol, the particles easily fell back into the bulk liquid in the acrylic pipe after a bit vibration given, whereas in experiments using ethanol, the particles did not easily fall back.

In addition, it was known that after the experiment, the floc that formed after the addition of alum decomposed back into smaller particles when flotation with electrolysis was done. This was due to the influence of the bubble. It was also known from the results of experiments, alum acted like a collector which selectively assisted Batik waste particles to attach to the bubble.

It could be seen that after the results of electrolysis after 12 minutes are green. This could be the influence of chromate produced during the electrolysis.

From the results, electrical conductivity showed a not so mean value. The electrical conductivity value would be used for velocity analysis and the diameter of bubbles generated from the electrolysis.

3.1 Bubble Velocity Analysis

Measurement results of the top-level swarm bubble were done. The total measured bubble was 30 for each voltage used. The value of the measurement uncertainty are 4.56% and 6.94% for voltage 15 V and 15 V liquid with ethanol, respectively, with 95% level of confidence. The maximum, minimum and average terminal velocity of bubbles produced with 15 V liquid without ethanol is 1.31, 0.85, and 1.04 cm/s, respectively. Meanwhile the maximum, minimum and average terminal velocity of bubbles produced with 15 V liquid with ethanol is 1.91, 0.86, and 1.27 cm/s, respectively.

Winarto (2011) explained that the addition of frother could produce the smaller bubbles. Smaller bubbles have a smaller velocity compared to the larger bubbles. In this study, the substance that acted as a frother was ethanol. In The result shows that the average velocity of the bubble with 15 V voltage liquid mixture plus ethanol is higher than 15 V liquid without ethanol. According to the explanation of Winarto (2011), the velocity of bubble using voltage 15 V liquid mixture plus ethanol should be lower. It was because bubbles generated by voltage 15 V liquid mixture plus ethanol was larger so the speed was higher. It may be because of the electrical conductivity value of liquid mixture plus ethanol was larger, I. e. 27.1 mS/cm, while electrical conductivity of liquid without ethanol was 23.2 mS/cm. The difference of the conductivity was because the liquid mixture with ethanol was in the lower level of the Batik waste storage. It has a larger conductivity due to the precipitation occurred in the storage.

3.2 Bubble Diameter Analysis

Bubble diameter data was grouped by classes and then the number of frequencies were counted. The value of the measurement uncertainty are 10.08% and 9.04% for voltage of 15 V and 15 V liquid plus ethanol, respectively, with 95% level of confidence. Each class was determined in accordance of its middle value and frequency then plotted and presented in Figure 7.

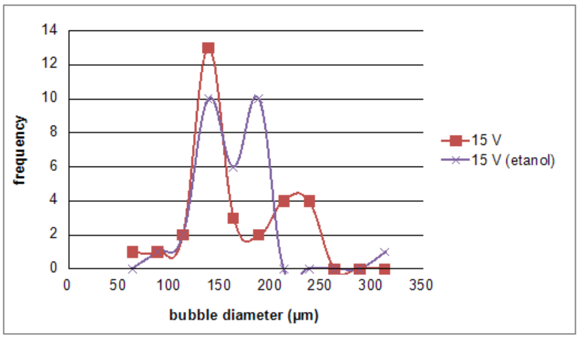

Figure 12. Frequency of bubble size generated

Figure 7 indicates that the voltage of 15 V, the dominant size is between 125 and 150 μm, but there were many bubbles produced with the size of 200 to 250 μm. It shows that the size of the bubble with 15 V liquid plus ethanol is dominated by bubble size between 125 and 200 μm. The average bubble size is 162.26 μm. This value is greater than the average size of the bubbles 15 V without ethanol. It clarifies the previous discussion, i. e. the bubble velocity of electrolysis using voltage of 15 V for liquid mixture plus ethanol had a greater value.

Al Shakarji, He, and Gregory (2011) explained that the final bubble size changes slightly with increasing current density. The existence of bubbles larger than the average bubble could be caused by merging of smaller bubbles. This could be due to bubbles generated are unstable.

3.3 Effect of Frother on The Percent Reduction of Color, Turbidity and TSS

From data processing, the addition of ethanol as frother into the liquid mixture could reduce its color and turbidity by 89.6% and 90.26%, respectively. This could be due to the addition of ethanol that made the bubbles more stable .

Percent reduction in TSS for liquid mixture without ethanol, the most effective voltage was 15 V, i. e. 95.96 %. With the addition of ethanol, percent reduction in TSS rise to 97.16%. Although the bubble was greater, the larger electrical conductivity caused the number of bubbles produced during the flotation process was also more. It increased the probability of collisions so that the flotation process more efficient, which in turn made the percent of TSS reduction for voltage 15 V liquid mixture plus ethanol was greater.

3.4 Velocity Analysis Based on Prediction of Stokes and Hadamard-Rybczynski

The derivation of the terminal velocity equation can be done by using two equations, i.e. the buoyancy force and drag force. It is known that by ignoring the gravity of the bubble (because the gas in the bubbles is very small), by the time the bubble reaches its terminal velocity, drag and buoyancy force has the same value:

$$F_B = F_d \qquad (1)$$

with the buoyancy force, $F_{B,}$ is formulated as :

$$F_B = \rho.V.g \qquad (2)$$

with ρ is the density of the fluid where the bubble moves (kg/m^3), V is the bubble volume (m^3) and g is the gravitaional acceleration (m/s^2). While the drag force F_d is formulated as:

$$F_d = C_D.1/2.\rho.U_T^2.A \qquad (3)$$

with C_D is drag coefficient and A is the surface area which in this case is the value of $\pi D^2/4$ with D is the diameter of the bubble. In this case the U_T is the terminal velocity of the bubble. By equating equations (2) and (3) the terminal velocity is:

$$U_T = \sqrt{\frac{4Dg}{3C_D}} \qquad (4)$$

Clift , Grace , and Weber (1978) explain that the value of C_D according to Hadamard-Rybczynski is :

$$C_D = \frac{8}{Re}\left(\frac{2+3\kappa}{1+\kappa}\right) \qquad (5)$$

where κ is the ratio of the viscosity of the fluid in the bubble with a viscosity of the fluid where the bubble moves. Navarra, Acuna and Finch (2009) explain that the terminal velocity of an ideal fluid (Newtonian) with a spherical form moving up another ideal fluid obtained by Rybczynski (1911) and independently by Hadamard (1911) for low Reynolds number (Re<1) based on Dukhin et. al. (1998), by taking the viscosity and the density of the water is much greater than air, the value of C_D according to Hadamard - Rybczynski is the same as 16/Re. Therefore, the terminal velocity in the water (according to Hadamard–Rybczynski) is:

$$U_{HR} = \frac{gd^2\rho}{12\mu} \qquad (6)$$

where g, d, ρ and μ is the gravitational acceleration (m/s^2) , bubble diameter (m), the density of the liquid where the bubbles move (kg/m^3) and viscosity of the liquid where the bubbles move (Pa.s), respectively. Clift, Grace, and (1978) explain that the value of C_D according to Stokes is the same as 24/Re. Therefore, the terminal velocity of a rigid spherical ball which is a classic result by Stokes (1851) is:

$$U_{ST} = \frac{gd^2\rho}{18\mu} \qquad (7)$$

To reduce these two equations, Re<1 is assumed to simplify the Navier-Stokes equations. According to Dukhin et. al. (1998) bubbles with the diameter ranging from 0.4 to 1.0 mm are in the intermediate regime (1<Re<100), but the equation (6) and (7) is still an important point in the comparison because of their simplicity, and because the analytic solutions can not be accessed at intermediate regime (Navarra, Acuna and Finch, 2009).

Comparisons have been made between the velocity and the diameter of the bubble based on the experimental results and the equations above. The dominant liquid in the mixture is the distilled water. Therefore, the viscosity values taken for equation (8), is the value of viscosity of water based on temperature:

$$\mu = a.10^{b/(T-c)} \quad (8)$$

whit a = 2,414.10-5 Pa.s, b = 247.8 K, c = 140.0 K and the temperature T is in K, the Kelvin scale. Equation (8) has a very good conformity with the data of Mott's experiments where the temperature ranges from 20°C to 30°C (Navarra, Acuna and Finch, 2009). This experiment was known that it was in the room temperature, i. e. 27.2°C, so the viscosity of the liquid used is 7.63 $x10^{-4}$ Pa.s. Based on the measurements, the density of the liquid used for experiments 15V is 947.85 kg/m^3, while the density of the liquid used for experiments 15 V liquid mixture with ethanol is 952 kg/m^3. Overall average velocity and average diameter of the all experimental data is taken and is put into the equation (6) and (7). The result is shown in Figure 8.

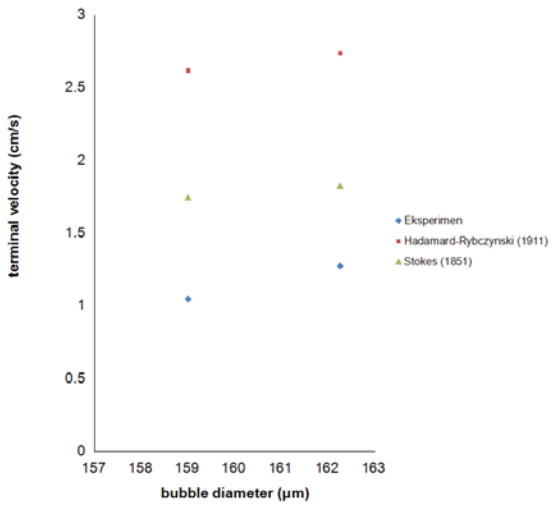

Figure 13. Graphics terminal velocity vs bubble diameter

From Figure 8, it can be seen that for bubbles produced by the voltage of 15V with and without ethanol, the value of the terminal velocity are below the predicted Stokes and must be below the predicted value of Hadamard-Rybczynski. This suggests that the average bubble moves slower than the predicted value by Stokes for solid ball moving in a liquid. It is as described by Navarra, Acuna and Finch (2009) and their research results this research, showing that there is a secondary mechanisms. Navarra, Acuna and Finch (2009) describe that as a bubble moves with a certain velocity, the bubble is deformed into an oblate spheroid, storing potential energy as well as Hooke's Law. When the diameter of the bubble is less than 1 mm, each deformation is linear and produces a symmetric oblate spheroid, as confirmed by Duineveld (1995) (Navarra, Acuna and Finch, 2009). In addition, Navarra, Acuna and Finch (2009) also explain that a spherical bubble experience shear stress when rising in a pulp, causing the stress gradient in the longitudinal direction. Frother on the bottom of the bubble tends to limit the compression, resulting in stagnant cap (stagnant cap can be regarded as an ultra-viscous). Therefore, the stress gradient combined (coupled) with frother concentration gradient resulting in the reduction of frother on the front or top and the increasing on the bottom or of the bubble. It causes the frother molecule adsorption on the top and the desorption on the bottom. A phenomenon due to the coupling of the concentration and the stress gradient is called the Marangoni effect (Navarra, Acuna and Finch, 2009).

3.5 Particle Size of Batik Dyeing Waste

Particle measurements have been carried out in the Test Materials Laboratory, Department of Metallurgy and Materials, Faculty of Engineering, University of Indonesia. The sample measured was the Batik waste sample of the liquid mixture as used in the experiments using liquid without ethanol. Measurements were performed using Scanning Electron Microscopy (SEM). The measurement results are shown in Figure 9.

Testing was done after the liquid mixture stirred for the particles floated in the liquid mixture surface. It can be seen in Figure 18 that the particle size is in the nano scale. Based on the measurement results it could be said that the flotation of Batik waste in this study can separate particles with the range in size from about 80 nm to about 105 nm.

4. Conclusion

Research on the dynamics of bubble flotation results with electrolysis has been done. Effect of ethanol on the bubble characteristics showed that the average diameter of the bubbles generated by the addition of ethanol in the liquid mixture was greater than that generated bubbles in liquid mixture without ethanol. Terminal velocity was also smaller than in the liquid mixture without ethanol. The addition of ethanol in the liquid mixture can rise the TSS reduction.

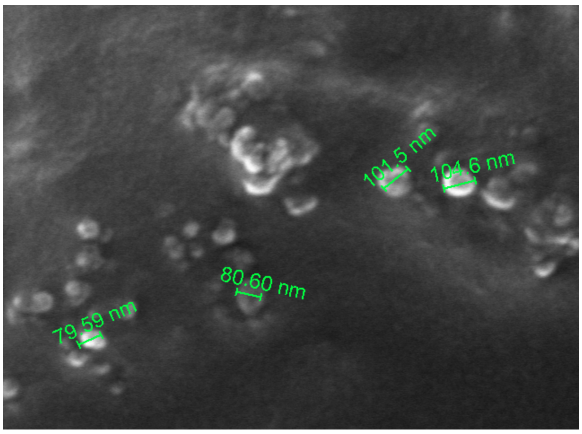

Figure 14. Particles of Batik waste

It can be inferred that the average terminal velocity of the bubbles are under the Stokes predictions. It suggests that a secondary mechanisms on the bubbles were occured.

DAFTAR PUSTAKA

Al Shakarji, R., He, Y., and Gregory, S. The sizing of oxygen bubbles in copper electrowinning. *Hydrometallurgy* Volume 109; (2011): pages 168–174

Basuki. Penyisihan zat organik pada air limbah industri Batik dengan fotokatalisis TiO_2. Surabaya: Environmental Engineering, Institut Teknologi Sepuluh November (2011)

Clift, R., Grace, J. R., and Weber, M. E. *Bubbles, Drops, and Particles*. New York: Academic Press, Inc. (1978)

Darmawanti, T. Pengolahan limbah cair industri Batik dengan metoda elektrokoagulasi menggunakan besi bekas sebagai elektroda", *Undergraduate Thesis*. Semarang: Chemistry FMIPA, Universitas Diponegoro (2009)

Hanotu, J., Bandulasena, HC., H., dan Zimmerman, W. B. Microflotation performance for algal separation. United Kingdom: Department of Chemical and Biological Engineering, University of Sheffield (2012)

Matis, K. A. *Flotation Science and Engineering*, CRC Press, Inc. (1995)

Navarra, A., Acuna, C., and Finch, J. A. Impact of frother on the terminal velocity of small bubbles. *International Journal of Mineral Processing*, Elsevier 91; (2009): 68–73

Rahmawati, N. I. Pengolahan limbah cair industri Batik dengan metoda elektrokoagulasi menggunakan seng sebagai elektroda. *Undergraduate Thesis*. Semarang: Chemistry FMIPA, Universitas Diponegoro (2009)

Riyanto. Penemuan teknik baru untuk pengolahan limbah Batik. Yogyakarta: Chemistry, Universitas Islam Indonesia (2011)

Setyaningsih, H. Pengolahan limbah Batik dengan proses kimia dan adsorpsi karbon aktif. *Master Thesis*. Jakarta: Graduate Program Universitas Indonesia (2007)

Shakir K., Elkafrawy, A. F., Ghoneimy, H.F., Elrab Beheir, S. G., Refaat, M. Removal of rhodamine B (a basic dye) and thoron (an acidic dye) from dilute aqueous solutions and wastewater simulants by ion flotation. *Journal International of Water Research*, Elsevier 44; (2010): 1449–1461

Winarto, F. E. W. Investigation of micro bubble for removal of suspended particle and enhancement of dissolved oxygen in poluted water. *Ph.D Thesis*. Perak: Mechanical

Engineering, Universiti Teknologi Petronas (2011)