

BUBBLE DYNAMICS OF BATIK DYEING WASTE SEPARATION USING FLOTATION

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ABSTRACT

Batik waste can increase water characteristics, such as turbidity, color and total suspended solids (TSS). Thus, an efficient technique for separating Batik from the liquid to decrease these characteristics is needed. The aim of the current study was to understand the results of flotation using electrolysis and to investigate the bubble characteristics that influence the results of the flotation of Batik waste. Flotation studies have been conducted using electrolysis to produce bubbles to separate batik synthetic dye from the liquid. Research conducted with 316L stainless steel electrodes, inside a 100 cm tall acrylic pipe with an inner diameter of 8.4 cm and a voltage variation of 10, 15 and 20 V. Batik waste was mixed with distilled water. Commercial alum powder [aluminum sulfate, $\text{Al}_2(\text{SO}_4)_3 \cdot 14\text{H}_2\text{O}$, that is 17% Al_2O_3] as the reagent was added to coagulate Batik waste in a ratio of 1 gram per 10 ml of Batik waste. The results showed that flotation of Batik waste can be used to separate Batik waste with the addition of alum. Alum was shown to be capable of acting as a collector in this type of waste separation. The results showed that flotation using electrolysis could be an effective method for reducing turbidity, color and TSS.

Keywords: Batik waste; Electroflotation; Electrolysis; Flotation; Microbubble

1. INTRODUCTION

Bubble flotation using electrolysis is also known as electroflotation or electrolytic flotation (Kyzas & Matis, 2016). Electroflotation is an alternative traditional technique for wastewater treatment that can be applied on a small, medium or large scale (Baghban et al., 2014; Choi et al., 2013; Shakir & Hussein, 2009; Kurniawan et al., 2006; Casqueira et al., 2006; Mouli et al., 2004). In the electroflotation method (EFM), ions or solid particles that are suspended or dissolved in a liquid phase are floated. The method uses the adhesion on very small bubbles of hydrogen and oxygen produced by electrolysis that moves upward in a flotation cell (da Mota et al., 2015). Flotation efficiency is affected by three parameters: the probability of collision between the bubbles and the particles, the probability the particles will stick to the surface of the bubbles and the probability the particles will be carried by the bubbles (Matis, 1995). The higher the probability, the higher the efficiency of the flotation. A higher number of smaller bubbles can increase the surface area per volume so that the efficiency can be improved (Coward et al., 2015). A microbubble is defined as bubble that has diameter between 50–200 μm (Deendarlianto et al., 2015). In wastewater treatment, devices to produce microbubbles are

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called microbubble generator (MBG) (Budhijanto et al., 2015). Electrolysis can also produce microbubbles (da Cruz et al., 2016; Lobo et al., 2016, Hashaikheh et al., 2014, Sarkar et al., 2010). Electrolysis produces hydrogen bubbles on the cathode and oxygen bubbles on the anode (Nanseu-Njiki et al., 2009; Saksono et al., 2012).

Meanwhile, Batik is an Indonesian cultural tradition. The rise in demand for Batik has increased the waste generated. Batik waste can degrade water quality by increasing its turbidity, color and total suspended solids (TSS). Several methods have been used by Indonesian researchers to solve this problem, such as coagulation, sedimentation, adsorption, filtration and flotation. The methods have been used in combination. For example, Setyaningsih (1995) combined coagulation, sedimentation and adsorption. The most frequently used method is adsorption (Darmawanti, 2009; Rahmawati, 2009; Basuki, 2011). Another method is flotation by electrolysis (Riyanto, 2011). However, Setyaningsih (1995) investigated several methods, including flotation, and found that the combination of coagulation, sedimentation and adsorption was more effective than coagulation and flotation. However, in the studies conducted by Riyanto (2011) and Setyaningsih (1995), a review of the flotation parameters did not include a discussion of the flotation method used. As a result, the cause of the low efficiency was hardly investigated in the study conducted by Setyaningsih (1995). The aim of the current study was to examine the result of flotation using electrolysis and to investigate the bubble characteristics that influence the flotation of the Batik waste.

2. METHODOLOGY

There are three main components in this experiment including apparatus, materials and experimental procedure that are explained as follow.

2.1. Apparatus

The equipment setup is shown in Figure 1.

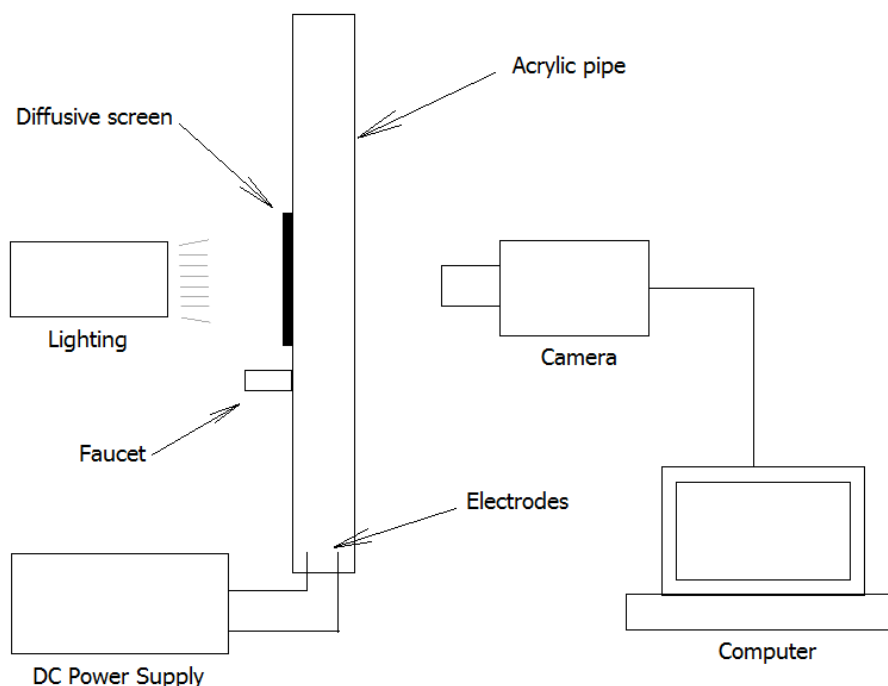


Figure 1 Experimental setup

The pipe was 100 cm long and 0.6 cm thick with an inside diameter of 8.4 cm. At about 25 cm

from the bottom of the pipe, a hole was drilled, and then a faucet was mounted and connected with a hose for sampling. Bubbles were generated with electrolysis. Thus, the bubbles were hydrogen and oxygen. The electrode used was 316L stainless steel. The 316L stainless steel was selected due to its resistance to wear in electrolysis. The electrodes consisted of five plates that had the same width and thickness, 5 cm and 0.1 cm, respectively. The electrodes were different lengths; two were 5.4 cm, two were 6.9 cm, and one was 8.2 cm. The electrode design was adapted to the size and the profile of the cross section of the round pipe. The plates were mounted parallel to the pipe on Styrofoam to maintain the position and the interplate distance. The interplate distance was about 1.5 cm. The middle (longest) plate and the edge (the two shortest) plates were the cathode with a total area of about 116 cm². The others were the anode with a total area of approximately 98 cm².

The camera was a complimentary metal oxide semiconductor (CMOS) camera with 12 megapixel resolution with a lens with 18–55 mm focal length. The bubbles generated by electrolysis were micro-sized (except bubbles that had merged with other bubbles). The lens made it hard to see the movement of bubbles. To overcome this issue, a technique called macro-photography was used. It uses an extension tube to create the magnification required to see the bubbles clearly. Extension tubes (ETs) were mounted between the lens and the camera. The fluid and the form of the pipe, which was cylindrical, could cause optical distortions. A ruler was inserted into the liquid to calibrate the distance and bubble diameter measurements.

The camera setup acted like a microscope. The lamps used to collect data on the velocity of the bubbles were two 9 W lamps. The backlighting technique was used by placing a diffusive screen on the back of the pipe in front of the camera to spread the light throughout the area. To collect data on the bubbles, high camera shutter speed was needed, which made the camera lack light. As a result, a 1500 W halogen lamp was used without an attached diffusive screen behind the acrylic pipe area. If the diffusive screen had been attached, only a black image would have been visible on the camera's liquid crystal display (LCD). The high-powered lamp generated heat. To overcome this issue, a transparent glass was placed between the pipe and the lamp. To keep the temperature constant, air was blown in the acrylic pipe near the lamp.

2.2. Materials

The acrylic pipe was filled with a mixture of 3500 ml of distilled water, 50 g of alum [aluminum sulfate, Al₂(SO₄)₃.14H₂O, that is 17% Al₂O₃], and 500 ml Batik staining waste produced by the Batik Production Center (Yogyakarta, Indonesia). A summary of the test conditions is presented in Table 1.

Table 1 Summary of the test conditions

Parameter	Remarks
Pipe material	Acrylic
Pipe dimension	8.4 cm diameter × 100 cm length
Electrodes	Stainless steel 316L
Anodes area	98 cm ²
Cathodes area	116 cm ²
Voltage variation	10, 15, 20 V
Batik waste volume	500 mL
Aquades volume	3500 mL
Alum mass	50 g
Flotation time variation	2, 4, 6, 8, 10, 12 mins
Total flotation time	12 mins
Sample taken	150 mL
Lamp/power	Halogen/1500 W for bubble size measurement Fluorescent/18 W for bubble velocity measurement
Diffusive screen	only for bubble velocity measurement

2.3. Experimental Procedure

The liquid mixture was poured into the pipe. Experiments were performed with voltage variations of 10, 15 and 20 V. The flotation experiment lasted for 2, 4, 6, 8, 10 and 12 min. Samples were collected to examine the turbidity, color and TSS. Each sample taken was 150 ml.

Measurements have shown that the pH of the liquid mixture after flotation is not very different from that before flotation, which is about 5. This was the effective pH value based on a flotation study using electrolysis carried out by Hanotu et al. (2012) for algae separation. In the present study, data were collected on the bubble characteristics (diameter and velocity) were collected after the Batik waste was floated. Thus, the liquid used for data collection of the bubble characteristics was the liquid of the flotation results.

3. RESULTS

3.1. Initial Experimental Results

According to Setyaningsih (1995), the synthetic dye waste from Batik staining can be directly floated without a reagent. Thus, the first experiment conducted was generating the bubbles to float the Batik waste directly. The sample was mixed with distilled water in a ratio of 2:3, respectively. Hydrochloric acid (HCl) was added to lower the pH value of the liquid mixture. The pH value was reduced to 8 for optimum results based on the study conducted by Setyaningsih (1995).

Experiments were carried out 2 times with electrolysis with 15 and 20 V, respectively, and 30 min flotation time. The results showed that with either 15 or 20 V there was no change in the fluid. This is shown in Figure 2 and Figure 3.

In Figures 2 and 3, from left to right are the initial samples, the sample after 5 min flotation, after 10 min flotation, after 15 min flotation, after 20 min flotation, the after 25 min flotation, and after 30 min flotation. No change was observed in the liquid. The analysis is discussed in the Discussion section.



Figure 2 Fluid samples after flotation with 15 V voltage

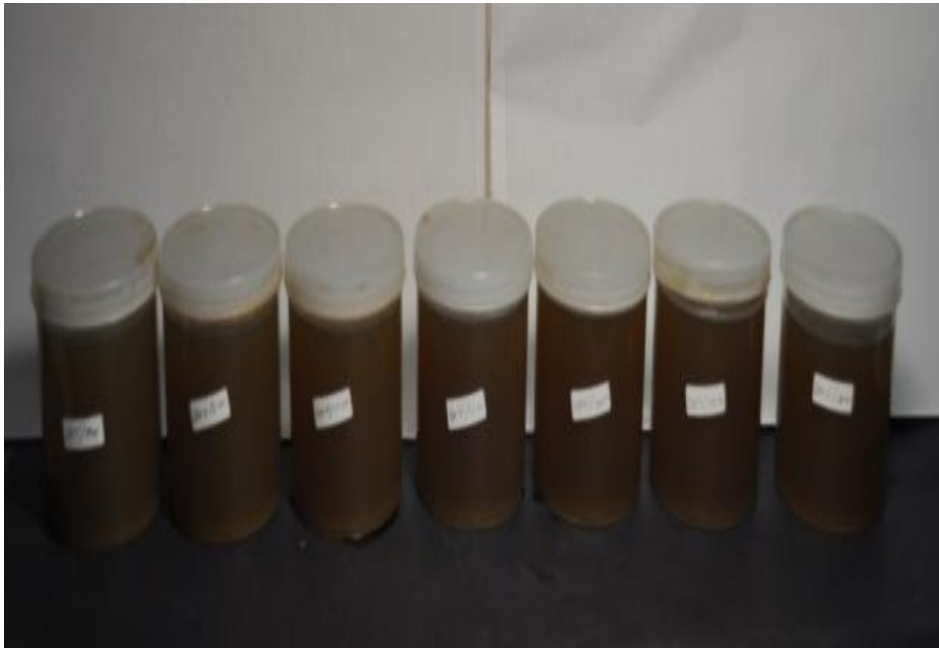


Figure 3 Fluid samples after flotation with 20 V voltage

3.2. The Influence of Voltage on Turbidity, Color and Total Suspended Solids

Figure 4, Figure 5, and Figure 6 show the flotation results for the samples using electrolysis. From left to the right are the sample before flotation, after 2 min flotation, after 4 min flotation, after 6 min flotation, after 8 min flotation, after 10 min flotation, and after 12 min flotation. There is no sample before flotation in Figures 5 and 6.



Figure 4 Samples of flotation with 10 V voltage



Figure 5 Samples of flotation with 15 V voltage



Figure 6 Samples of flotation with 20 V voltage

The decreases in the three parameters for each applied voltage are presented in Table 2.

Table 2 Flotation results

Voltage	Percent reduction of:		
	Turbidity	Color	Total suspended solids (TSS)
10 V	69.62%	83.28%	93.96%
15 V	63.80%	78.56%	95.98%
20 V	56.29%	72.06%	93.96%

4. DISCUSSION

Setyaningsih (1995) suggested that Batik waste can be floated directly without the addition of a reagent. However, in that initial study, the Batik particles were not floated. The Batik waste particles may have had low hydrophobicity, which means it did not easily stick to the bubbles. In additional experiments, the addition of alum made the Batik waste particles lifted. Figure 7 shows Batik waste inside an acrylic pipe before and after flotation.



Figure 7 Batik waste before (left) and after (right) 10 V flotation

For the samples, the turbidity, color and TSS reduction (or which were lifted) for the 10 V voltage were 69.62%, 83.28% and 93.96%, respectively. The turbidity, color and TSS reduction for the 15 V voltage were 63.8%, 78.56% and 95.98%, respectively. The turbidity, color and TSS reduction for the 20 V voltage were 56.9%, 72.06% and 93.96%. The highest decrease in color and turbidity occurred when the 10 V voltage was used. The bubbles generated using 10 V voltage were smaller. The smaller the bubble, the lower the terminal velocity. Additional

discussion is provided in the analysis of the bubble characteristics section. However, the TSS decrease was slightly different as the highest reduction was reached with 15 V voltage.

Before flotation, the particles from alum adhered to the Batik waste particles which made the particles combined into larger sizes (floc). Nurrohman (2012) showed that the bubble terminal velocity in drinking water with alum was lower than in drinking water without alum. This result suggests that the alum particles attached to the bubbles and prevented the bubbles' internal circulation. The alum had high hydrophobicity that made it easy for the alum to stick to the bubble. Thus, in the current study, alum acted as a collector. It seems to have "two arms." One "holds" the bubbles, and the other "holds" the Batik waste particles, which lifts and separates them from the liquid. The result may suggest the cause of the low efficiency in the study conducted by Setyaningsih (1995).

In addition, after the flotation, the flocs separated back into smaller particles when the flotation was completed. The bubble lifted the alum along with the Batik waste particles. The water became a little green after 12 min flotation because of the chromate produced during electrolysis.

4.1. Bubble Velocity Analysis

Measurement results of the top-level swarm bubble are shown in Figure 8. Thirty bubbles were measured for each voltage used. The upper limit of the line is the value of the bubble with the highest velocity, and the lower limit of the line is the value of the bubble with the lowest velocity. The uncertainty was 6.70%, 4.56% and 8.62% for the 10, 15 and 20 V voltage, respectively. The confidence level was 95%.

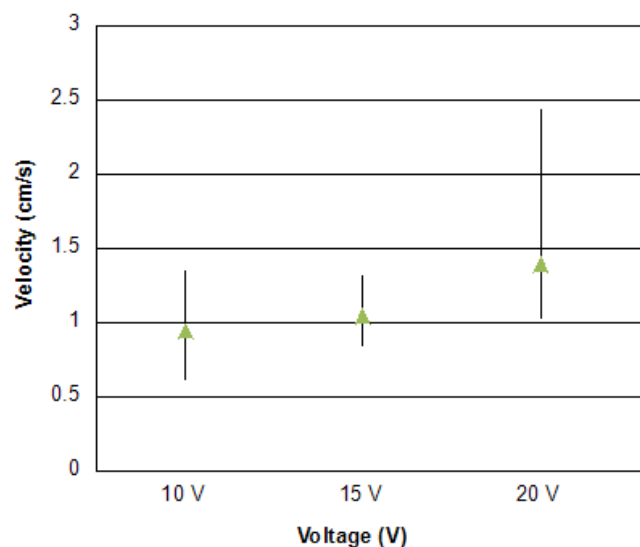


Figure 8 Comparison of bubble velocity with different applied voltages

Figure 8 shows that the average velocity of the bubbles are larger as the voltage used increases. The higher the voltage applied, the bigger the bubble diameter. The bubble diameter analysis is presented in the next section. All the bubble velocities were between 0.5 and 2.5 cm/s. These results are in accordance with experimental results found by Haapala et al. (2010), who conducted flotation with a sintered aerator to produce microbubbles in the presence of carboxymethyl cellulose (CMC), n-butanol and wood fibers. The authors found that the velocity of microbubbles between 100 μm and 300 μm ranged from 0.3 cm/s to 2.5 cm/s.

4.2. Bubble Diameter Analysis

Figure 9 shows bubbles rising inside the acrylic pipe. The distance between the two lines is 1 mm. The bubble diameter data were grouped by classes, and then the bubbles were counted. The measurement uncertainty was 5.16%, 10.08% and 8.82% for the 10, 15 and 20 V voltage, respectively. The confidence level was 95%. For each applied voltage, the measured bubbles were grouped into 11 classes: 50–75, 75–100, 100–125, 125–150, 150–175, 175–200, 200–225, 225–250, 250–275, 275–300 and 300–325 μm . The median of each class was then plotted against the frequency. The results are presented in Figure 10.

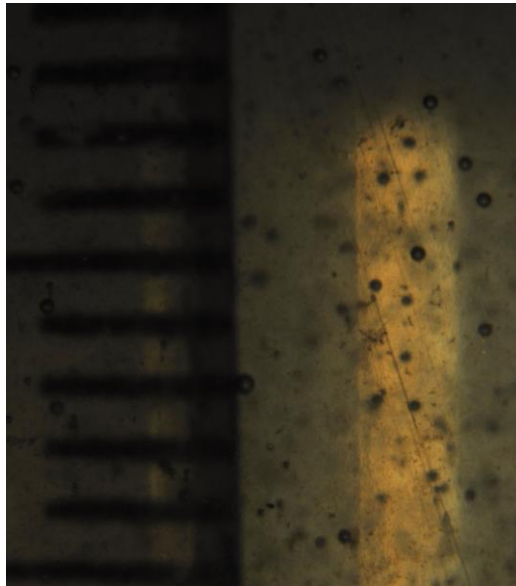


Figure 9 Bubbles rising inside the acrylic pipe

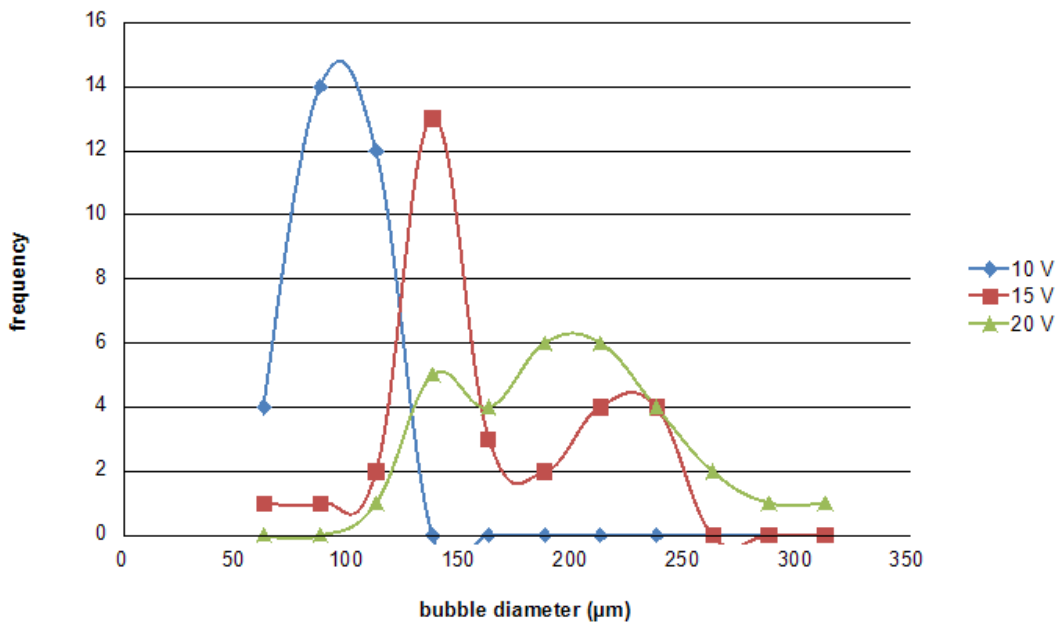


Figure 10 Frequency of bubble size generated

Figure 10 indicates that the 10 V voltage had a dominant bubble size of 75 to 120 μm . For the 15 V voltage, the dominant bubble was between 125 and 150 μm , but some bubbles were 200

to 250 μm . For the 20 V voltage, the dominant bubble was between 125 and 250 μm . The average bubble size for the 10, 15 and 20 V voltage was 93.63, 158.99 and 197.28 μm , respectively. The higher the voltage used, the larger the average bubble generated. This result clarifies the previous discussion which said that the higher the bubble diameter, the higher the bubble terminal velocity. In general, the method used produced microbubbles which is in accordance with other experiments by da Cruz et al. (2016), Lobo et al. (2016), Hashaikh et al. (2014) and Sarkar et al. (2010). Al Shakarji et al. (2011) explained that the final bubble size changes slightly with increasing current density. Thus, bubbles larger than the average bubble could exist because smaller bubbles unify, and the bubbles generated were unstable.

One of the biggest bubble in the current study had a diameter of 250 μm , and the velocity for all bubbles was below 2.5 cm/s whereas Huang et al. (2011) found that bubbles with a diameter of 250 μm had a velocity of 3.4 cm/s in pure water. Mei et al. (1994) predicted that bubbles with a diameter of 250 μm would have a velocity higher than 2.5 cm/s in pure water. It may be because the alum surfactant and the Batik particles disturbed the bubbles' internal circulation. The bubbles tended to have an immobile surface. Fan and Tsuchiya (1990) confirmed that surfactants can contaminate the bubbles, which creates an immobile surface. Bubbles with an immobile surface have lower terminal velocity compared to bubbles with a mobile surface as indicated by Clift et al. (1978). Manica et al. (2014) stated that, in ultra-clean water, bubbles attain larger velocity which is related to a mobile (stress free) boundary condition at the bubble surface whereas the presence of surfactants makes the surface immobile and results in lower velocity.

4.3. Batik Waste Particle Size

Measurements were performed using scanning electron microscopy (SEM). SEM is used to analyze the surface of a sample by firing electrons with high energy. The electrons interact with atoms on the sample so that signals that contain surface topography information are produced. The measurement results are shown in Figure 11.

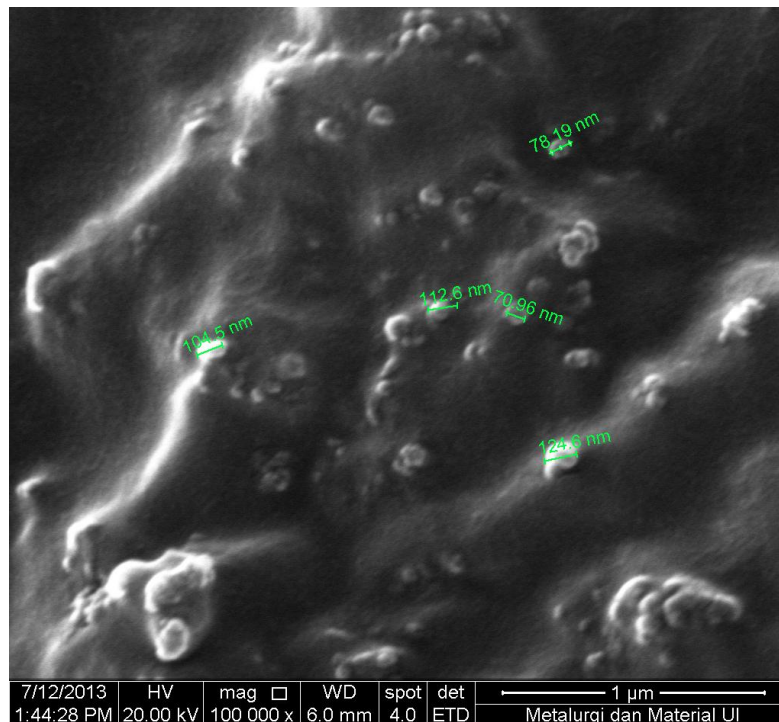


Figure 11 Batik waste particle size

Testing was performed after the liquid mixture was stirred for the particles floating in the liquid mixture surface. The particle size was in the nano scale. Thus, the flotation of Batik waste in the current study separated particles in the range from about 70 nm to about 120 nm.

5. CONCLUSION

The important result of the current study is that Batik waste cannot be floated directly using bubbles. A reagent is needed for the Batik waste to easily attach to the bubbles. Alum can be used. Flotation by electrolysis is an effective method for separating typical Batik waste. The most efficient color and turbidity reduction occurred with the lowest voltage, 10 V. However, the decrease in TSS reached more than 90% for all voltages applied. Thus, bubble dynamics had an important role in the separation process.

6. REFERENCES

- Al Shakarji, R., He, Y., Gregory, S., 2011. The Sizing of Oxygen Bubbles in Copper Electrowinning. *Hydrometallurgy*, Volume 109, pp. 168–174
- Baghban, E., Mehrabani-Zeinabad, A., Mohed, A., 2014. The Effects of Operational Parameters on the Electrochemical Removal of Cadmium Ion from Dilute Aqueous Solutions. *Hydrometallurgy*, Volume 149, pp. 97–105
- Basuki, 2011. *Penyisihan Zat Organik pada Air Limbah Industri Batik dengan Fotokatalisis TiO_2* . Surabaya: Environmental Engineering, Sepuluh November Institute of Technology [in Bahasa]
- Budhijanto, W., Deendarlianto, Kristiyani, H., Satriawan, D., 2015. Enhancement of Aerobic Wastewater Treatment by the Application of Attached Growth Microorganisms and Microbubble Generator. *International Journal of Technology*, Volume 6(7), pp. 1101–1109
- Casqueira, R.G., Torem, M.L., Koler, H.M., 2006. The Removal of Zinc from Liquid Streams by Electroflotation. *Minerals Engineering*, Volume 19(13), 1388–1392
- Choi, J., Shim, S., Yoon, J., 2013. Design and Operating Parameters Affecting an Electrochlorination System. *Journal of Industrial and Engineering Chemistry*, Volume 19(1), pp. 215–219
- Clift, M., Grace, R., Weber, J.R., 1978. *Bubbles, Drops and Particles*. Academic Press, New York, pp. 169–202
- Coward, T., Lee, J.G.M., Caldwell, G.S., 2015. The Effect of Bubble Size on the Efficiency and Economics of Harvesting Microalgae by Foam Flotation. *Journal of Applied Phycology*, Volume 27(2), pp. 733–742
- da Cruz, S.G., Dutra, A.J.B., Monte, M.B.M., 2016. The Influence of Some Parameters on Bubble Average Diameter in an Electroflotation Cell by Laser Diffraction Method. *Journal of Environmental Chemical Engineering*, In Press, Corrected Proof. Available online 17 May 2016
- da Mota, I.d-O., de Castro, J.A., Casqueira, R.d-G., de Oliveira Junior, A.G., 2015. Study of Electroflotation Method for Treatment of Wastewater from Washing Soil Contaminated by Heavy Metals. *Journal of Materials Research and Technology*, Volume 4(2), pp. 109–113
- Darmawanti, T., 2009. *Pengolahan Limbah Cair Industri Batik dengan Metoda Elektrokoagulasi Menggunakan Besi Bekas sebagai Elektroda*. Undergraduate thesis. Semarang: Chemistry Department, Diponegoro University [in Bahasa]
- Deendarlianto, Wiratni, Tontowi, A.E., Indarto, Iriawan, A.G.W., 2015. The Implementation of a Developed Microbubble Generator on the Aerobic Wastewater Treatment. *International Journal of Technology*, Volume 6(6), pp. 924–930

- Fan, L.-S., Tsuchiya, K., 1990. *Bubble Wake Dynamics in Liquids and Liquid-solid Dispersions*. Butterworth-Heinmann, Boston
- Haapala, A., Honkanen, M., Liimatainen, H., Stoor, T., Niinimäki, J., 2010. Hydrodynamic Drag and Rise Velocity of Microbubbles in Papermaking Process Waters. *Chemical Engineering Journal*, Volume 162(3), pp. 956–964
- Hanotu, J., Bandulasena, H.C.H., Zimmerman, W.B., (2012), Microflotation performance for algal separation. Department of Chemical and Biological Engineering, University of Sheffield
- Hashaikh, R., Laila, B.S., Kochkodan, V., Hilal, N., 2014. A Novel in Situ Membrane Cleaning Method using Periodic Electrolysis. *Journal of Membrane Science*, Volume 471, pp. 149–154
- Huang, Z., Legendre, D., Guiraud, P., 2011. A New Experimental Method for Determining Particle Capture Efficiency in Flotation. *Chemical Engineering Science*, Volume 66(5), pp. 982–997
- Kurniawan, T.A., Chan, G.Y.S., Lo, W.-H., Babel, S., 2006. Physico-chemical Treatment Techniques of Wastewater Laden with Heavy Metals. *Chemical Engineering Journal*, Volume 118(1-2), pp. 83–98
- Kyzas, G.Z., Matis K.A., 2016. Electroflotation Process: A Review. *Journal of Molecular Liquids*, Volume 220, pp. 657–664
- Lobo, F.L., Wang, H., Huggins, T., Rosenblum, J., Linden, K.G., Ren, Z.J., 2016. Low-energy Hydraulic Fracturing Wastewater Treatment via AC Powered Electrocoagulation with Biochar. *Journal of Hazardous Materials*, Volume 309, pp. 180–184
- Manica, R., Hendrix, M.H.W., Gupta, R., Klaseboer, E., Ohl, C.-D., Chan, D.Y.C., 2014. Modelling Bubble Rise and Interaction with a Glass Surface. *Applied Mathematical Modeling*, Volume 38(17-18), pp. 4249–4261
- Matis, K.A., 1995. *Flotation Science and Engineering*, CRC Press, Inc., New York
- Mei, R., Klausner, J.F., Lawrence, C.J., 1994. A Note on the History Force on a Spherical Bubble at Finite Reynolds Number. *Physics Fluids*, Volume 6(1), pp. 418–420
- Mouli, P.C., Mohan, S.V., Reddy, S.J., 2004. Electrochemical Processes for the Remediation of Wastewater and Contaminated Soil: Emerging Technology. *Journal of Scientific & Industrial Research*, Volume 63(1), pp. 11–19
- Nanseu-Njiki, C.P., Tchamango, S.R., Ngom, P.C., Darchen, A., Ngameni, E., 2009. Mercury(II) Removal from Water by Electrocoagulation using Aluminium and Iron Electrodes. *Journal of Hazardous Materials*, Volume 168(2-3), pp. 1430–1436
- Nurrohman, 2012. *Analisis Karakteristik Small Bubble pada Larutan Tawas*. Undergraduate thesis. Depok: Mechanical Engineering Department, University of Indonesia [in Bahasa]
- Rahmawati, N.I., 2009. *Pengolahan Limbah Cair Industri Batik dengan Metoda Elektrokoagulasi menggunakan Seng sebagai Elektroda*. Undergraduate thesis. Semarang: Chemistry Department, Diponegoro University [in Bahasa]
- Riyanto, 2011. *Penemuan Teknik Baru untuk Pengolahan Limbah Batik*. Islamic University of Indonesia, Yogyakarta [in Bahasa]
- Saksono, N., Ariawan, B., Bismo, S., 2012. Hydrogen Production System using Non-thermal Plasma Electrolysis in Glycerol-KOH Solution. *International Journal of Tehcnology*, Volume 3(1), pp. 8–15
- Sarkar, M.S.K.A., Evans, G.M., Donne, S.W., 2010. Bubble Size Measurement in Electroflotation. *Minerals Engineering*, Volume 23(11-13), pp. 1058–1065
- Setyaningsih, H., 1995. *Pengolahan Limbah Batik dengan Proses Kimia dan Adsorpsi Karbon Aktif*. Master Thesis. Jakarta: Graduate Program, University of Indonesia [in Bahasa]

Shakir, I.K., Hussein, B.I., 2009. Lead Removal from Industrial Wastewater by Electrocoagulation Process. *Iraqi Journal of Chemical and Petroleum Engineering*, Volume 10(2), pp. 35–42