Combined Process of Ozonation and Membrane Processes to treat Wastewater from Batik Industry

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Abstract. Batik is a cultural heritage which was handed down to her craftsmen in Indonesia, even now it is recognized by UNESCO as one of the world heritages. However, the batik industry is also known as an industry that discharges a lot of wastewater so that the processing of batik waste becomes very important to prevent surface water pollution from batik wastewater. Therefore, this study aims to examine the performance of polypropylene (PP) fibers-based membrane module in wastewater treatment from the batik industry. The batik wastewater was prior treated through a combination of ozonation (O₃) and flocculation, and then ultra-filtered by a PP membrane module at various trans membrane pressures (TMP). Conductivity, total suspended solids (TSS), chemical oxygen demand (COD) and color (Pt/Co) were the observed variables to examine the performance of the batik wastewater treatment process. The experimental results show that TSS, COD and Pt/Co can be reduced by about 99.8%, 24% and 57%, respectively. Meanwhile, the permeate fluxes from the membrane module were 141.3, 182.0, 243.9, 264.7 and 290.88 L.m⁻².h⁻¹ at TMP of 3, 5, 7, 9 and 11 bars, respectively.

Keywords: Batik wastewater, COD, color, TSS, membrane.

1. Introduction

The development of the textile industry in Indonesia is experiencing quite rapid progress, especially in the regions of Central Java and East Java, one of which is batik [1]. Batik is a cultural heritage which was handed down to her craftsmen in Indonesia, even now it is recognized by UNESCO as one of the world heritages. This development certainly has a positive impact on the regional economy and society in the region. However, on the other hand, the batik industry generates a large amount of harmful waste. In the batik industry, the waste generated from the washing process requires water as a medium in large quantities. This process contains large waste water and contains residual color, high BOD, high oil content and high hazardous and toxic materials waste which far exceeds the water quality standard based on the Decree of the State Minister for Population and Environment No. KEP-51/MENLH/10/1995. [1, 2]. Since most of the waste is in liquid form, it is generally discharged to waterways. Therefore, treating of batik waste becomes very important to prevent surface water pollution from batik wastewater.

There have been many methods used to treat batik liquid waste to reduce the dangerous impact for the environment. From processed by coagulation, precipitation, filtering [3-6] and absorption [4], using
chemical, aerobic and anaerobic biology [6, 7], sewage treatment system uses a combination of alum and biological physics [8], artificial wetland technology [5], using TiO₂-Dopan-N Photocatalyst with Sunlight as an energy source [9], Using TiO₂ Nanoparticles Coated on the Surface of Plastic Sheet [10]. Utilization of Pb and PbO₂ from lead storage battery waste using electrochemical method [11], indirect oxidation process with electrocatalytic reactor [12], using CA-PS composite membrane as a filter with dead end membrane reactor [13], using cassava cellulose acetate membrane synthesis for microfiltration of Fe [14], utilization of zeolite and silica as membrane filtration materials [15] and so many others, which achieve varied results. Various methods and its combinations are carried out to produce more effective batik wastewater treatment. In this research is using a combination of ozonation and flocculation, and then ultra-filtration processes with polypropylene membranes. This study intent to examine the performance of polypropylene (PP) fibres-based membrane module in wastewater treatment from the batik industry. Ozonation is a process generally performed in water and wastewater treatment for disinfection along with oxidation of micropollutants and natural organic matter [16]. Batik wastewater is pre-treated with the combination processes of Ozonation and flocculation to prevent fouling when effluent is filtered using polypropylene membrane.

2. Materials and Methods

2.1 Materials

Material used for the Ozonation process are Ozone, Ozone Reagent from Merck and H₂SO₄ 2M. Material used for filtration process is polypropylene fiber membrane. For analyses and general purposes, are used distilled water, COD Reagent from HACH cat. 2125925, KI, Na₂S₂O₃, KMnO₄ and Poly Aluminium Chloride (PAC).

| Parameter                        | Information                  |
|----------------------------------|------------------------------|
| Inside diameter of each fibre    | 235 μm = 2.35 x 10⁻⁴ m       |
| Thickness of each fiber          | 145 μm = 1.45 x 10⁻⁴ m       |
| Outside diameter of each fiber   | 525 μm = 5.25 x 10⁻⁴ m       |
| Material                         | Polypropylene                |
| N (amount of fiber)              | 1.200 pcs                    |
| Effective length                 | 25 cm = 0.25 m               |
| Surface Area                     | 4,125 x 10⁻⁴ m               |

Instrumentations used for the Ozonation process are Commercial household appliance Ozonator (X-Troy and Hanaco), Packed Bed Reactors (PBR), venturi injector (Mazzei), wastewater reservoir, diaphragm pump, hose, stirrer (IKA), Buchner Funnel Porcelain and magnetic bar. Instrumentations used for the filtration process are polypropylene membrane module, reverse osmosis pumps, flow meter, three-way valve, pressure gauge, container (feed, permeate and retentate), analytical balance (OHAUS Pioneer) and stopwatch. For analyses and tests, are used glasses apparatus, heating block digester (DRB 200), Ultraviolet Visible Spectrophotometer (BEL UV-MS1 single beam) with vial and cuvette, Conductivity meter, Colorimeter (HACH DR890), test tubes and pH meter.

2.2 Batik Wastewater Preparation

The batik wastewater sample used in this research was taken from Central Java area, particularly in District area of Tegal. The sample is taken randomly and mixed, considering there is no specific channel
that separates batik wastewater from other wastes in the existing small industry. Since the batik wastewater is concentrated, it is diluted with distilled water by the ratio of waste and distilled water are 1 and 4, respectively. To overcome the bigger particles and suspension solids, which may lead to membrane fouling or even module damage, the diluted sample is pre-filtered by vacuum filter using Buchner Funnel Porcelain with Whatman filter paper.

The initial pH of the diluted and pre-filtered sample is around 8. Then, a series of various pH (4, 6, 8 and 10) of the sample were conducted with a jar test using PAC, including in its original pH. Adjusting the pH of the sample is using H₂SO₄ 2M. The optimum pH was obtained at 4, where the test result for Conductivity, COD, TSS and Pt/Co are given the best results compare than other varied pH value.

2.3 Ozonation and Flocculation Processes

After series of flocculation experiments using PAC in the jar test to determine which pH and PAC concentration that give best result in terms of Batik wastewater quality such as Conductivity, TSS, Pt/Co and COD, the determined pH (4) and PAC concentration (500 ppm) are conducted to get the bigger volume of pre-treated Batik wastewater to be fed for Ozone and Membrane Processes.

4 liters of the sample is poured into reactor and adjusted the pH to 4 with addition of H₂SO₄ 2M. Then, Ozone from the Ozonator is streamed into the reactor through the injector with flowrate of 1.19 liter per minute with simultaneously turning on the stirrer with a fast-stirring of 120 rpm for 4 minutes. After 4 minutes, the Ozonator is turned off. The stirrer is adjusted to 40 rpm, then a slow-stirring is carried out for 10 minutes. Subsequently, the solution is settled for about 30 minutes and ready for filtration process along with collected some for Conductivity, TSS, Pt/Co and COD tests.

2.4 Filtration Process by Polypropylene Membrane

5 liters of the sample produced and mixed from several Ozonation Process with same condition is then placed at feed container of the membrane unit equipment, which consist of 2 reverse osmosis (RO) pumps, flow meter, three-way valve, pressure gauge, container (feed, permeate and retentate) and polypropylene membrane module itself. There was additional analytical balance to weigh permeate produced at certain time.

The experiment starts with turning on both RO pumps and regulate the Transmembrane Pressure (TMP) to desired value. Permeate and retentate are start to produced. The experiments were performed in semi recycle mode of filtration, where the retentate is continuously recirculated into the feed container. Permeate is first collected to a temporary container. After the TMP is stable at desired value, permeate is then collected to another container on the analytical balance while time is started to record simultaneously. Weight gain of permeate is recorded for a minute. After an exact minute, the timer is stopped and the pumps are switched off. Some permeate sample is collected for water quality analyses, the rest of it is remixed with the sample in the feed container. The experiment is repeated 2 more times with the same value of TMP and same time of recorded weight gain of permeate.

The experiments at 5 values of TMPs (3, 5, 7, 9 and 11 bars) were carried out to observe performance of membrane at different value of TMPs. The filtration performance was observed for both permeate flux and permeate quality. The permeate flux is calculated refer to equation (1) below:

$$ J = \frac{V}{A \times \Delta t} $$ (1)

While the permeate quality is calculated based on the analyses results of the parameter such as TSS, Pt/Co and COD to determine the removal efficiency of the parameter. Removal efficiency is calculated to identify how far the process could remove/reduce those parameters, using the following equation (2):
Removal Efficiency (%) = \frac{(Value\ in - Value\ out)}{Value\ in} \times 100\% \quad (2)

3. Results and Discussion

3.1 Wastewater Characterization

Effluents from the batik industry contain large amounts of organic pollutants, which lead to 17-20% of industrial freshwater pollution. It has a high pH value (typically between 7 and 9), dark color, and a high content of Chemical Oxygen Demand (150-12000 mg/L), Biochemical Oxygen Demand (80-6000 mg/L), and Total Organic Carbon (2900 and 3100 mg/L) depending on the processes [17].

Corresponding to the references above, the analysis of Batik wastewater obtained result as follow:

Table 2. Characterization of Batik wastewater

| Parameter                        | Result       |
|----------------------------------|--------------|
| pH                               | 8.2          |
| Conductivity                     | 4.41 μS/cm   |
| Total Suspended Solids (TSS)     | 1,745 mg/L   |
| Pt/Co                            | 22,225.95 TCU|
| Chemical Oxygen Demand (COD)     | 4,900.45 mg/L|
| Appearance                       | Dark Green   |

The nature of textile effluents characterized by alkaline conditions [18], as for as we can see from table 2, the pH of the wastewater is 8.2. Batik wastewater is commonly in alkaline condition. This is due to the use of organic dyes compounds which are mostly alkaline. Moreover, in coloring, this compound is only used about 5% while the remaining 95% will be discarded as waste [8]. The appearance is dark green, in line with the high Pt / Co values.

3.2 Permeability of Distilled Water

As mentioned earlier, distilled water permeability was performed three times before set of experiments are conducted, which also three times for each varied TMP. Permeate produced from the membrane module is weighed for 1 minute. Weight gain every 5 seconds are recorded as follow:

Table 3. Weight Gain of Distilled Water Permeability Test

| Time (second) | 1st attempt | 2nd attempt | 3rd attempt | Mean   |
|---------------|-------------|-------------|-------------|--------|
| 5             | 85.88       | 83.09       | 83.01       | 83.99  |
| 10            | 161.61      | 151.96      | 152.67      | 155.41 |
| 15            | 231.4       | 217.7       | 218.16      | 222.42 |
| 20            | 306.63      | 294.5       | 285.48      | 295.54 |
| 25            | 381.41      | 367.6       | 351.08      | 366.70 |
| 30            | 452.43      | 434.93      | 420.73      | 436.03 |
| 35            | 524.1       | 504.14      | 482.83      | 503.69 |
| 40            | 598.48      | 575.25      | 555.83      | 576.52 |
| 45            | 673.05      | 647.48      | 624.63      | 648.39 |
| 50            | 739.17      | 714.55      | 691.66      | 715.13 |
| 55            | 816.71      | 786.3       | 758.4       | 787.14 |
| 60            | 882.02      | 845.29      | 823.96      | 850.42 |
The weighing process is accomplished by weighing the permeate coming out of the membrane module into the permeate container located on the analytical balance (OHAUS Pioneer) which connected to Computer to record the weight gain every certain time. The following curve in Figure 1 shows the relation between average weight gain versus time. It can be seen that the line is almost linear with $R^2$ equal to 0.9999.

By using Equation 1, membrane properties from Table 1 and average weight of distilled water as permeate from Table 3, flux value of distilled water can be calculated. The calculation result for the flux is $429.45 \text{ L.m}^{-2}\text{ h}^{-1}$.

### 3.3 Permeability of Batik Wastewater

Similar to permeability of distilled water in terms of procedure and attempt number, set of filtrations using membrane module were conducted three times per various TMPs i.e. 3, 5, 7, 9 and 11 bars. Permeate produced from the membrane module for each attempt at each TMP, are weighed for 1 minute. Average weight gains every 5 seconds for all various TMPs are recorded as follow:

| Time (second) | 3 bars | 5 bars | 7 bars | 9 bars | 11 bars |
|---------------|--------|--------|--------|--------|---------|
| 5             | 27.64  | 37.66  | 50.87  | 53.75  | 57.28   |
| 10            | 50.96  | 66.74  | 88.77  | 95.93  | 106.32  |
| 15            | 74.26  | 96.15  | 128.89 | 140.33 | 154.42  |
| 20            | 97.09  | 124.55 | 167.67 | 183.83 | 200.94  |
| 25            | 120.57 | 153.68 | 207.99 | 225.22 | 249.78  |
| 30            | 143.61 | 182.84 | 245.15 | 267.77 | 295.05  |
| 35            | 166.71 | 212.35 | 282.31 | 308.85 | 340.21  |
| 40            | 189.67 | 240.53 | 322.31 | 353.63 | 388.01  |
| 45            | 212.18 | 270.57 | 361.07 | 392.44 | 434.04  |
| 50            | 234.89 | 301.16 | 399.62 | 435.91 | 479.68  |
| 55            | 257.42 | 327.42 | 440.28 | 477.12 | 526.85  |
| 60            | 279.12 | 355.97 | 476.88 | 516.37 | 569.75  |
As of Figure 2 shows above, almost all lines are liner with $R^2 = 0.9999$. There is even 1 data value has $R^2$ equal to 1, which is at TMP 7 bars. This explains that the polypropylene membrane used in the experiment, working properly and shows performance consistency, either using distilled water or Batik wastewater as the feed.

From the data above, using Equation 1 and membrane properties from Table 1, fluxes value of various TMPs Batik Wastewater can be calculated. The result of calculation on every 5 seconds at each various TMPs are display in the following Figure 3.

The curves show similar trend for all various TMPs for recorded 5 seconds to 60 seconds. It clarifies that the polypropylene membrane used in this experiment still shows the same function and performance during the attempts. If fluxes of each various TMP compared between permeate produced at 5 seconds with 60 seconds, there are quite similar performance percentage for all various TMPs, around 80%. The results are 84.15, 78.77, 78.12, 80.05 and 82.89% for 3, 5, 7, 9 and 11 bars respectively.
The fluxes value for various TMPs at 3, 5, 7, 9 and 11 bars are 141.31, 182.03, 243.91, 264.75 and 290.88 L.m$^{-2}$.h$^{-1}$ respectively. From the data and curves above, as pressure increases, the value of the flux will also rise. Obviously, since the pressure is getting higher, the flowrate of feed sucked into membrane inlet will also increase which led to higher output produced, both to permeate as well as retentate container.

3.4 Treated Batik Wastewater Characterization

Combined process of Ozonation and Membrane Processes are conducted sequentially. Wastewater quality tests are performed to raw sample, after pre-treated (ozonation process) and after filtered (membrane process). The results are shown below in Table 5.

Table 5. Treated Batik Wastewater Characterization

| Parameter         | Raw   | Pre-treated (Ozonation) | Treated (Membrane Process) |
|-------------------|-------|-------------------------|----------------------------|
| pH                | 8.2   | 4.28                    | 4.27 4.27 4.28 4.28 4.28 4.27 |
| Conductivity (μS/cm) | 4,41  | 2,91                    | 2,92 3,04 3,03 3 3,02    |
| TSS (mg/L)       | 1745  | 39                      | 1 2 4 5 3              |
| Pt/Co (TCU)      | 22225,95 | 179,14                | 3,44 1,91 3,44 3,44 3,44 |
| COD (mg/L)       | 4900,45 | 561,33              | 428,26 425,28 429,35 429,08 431,25 |

From Table 5 above and figure 5 below, pH value of wastewater is intentionally reduced from 8.2 (as raw wastewater) to 4.28 (as outcome from pre-treated/ozonation), where the optimum pH for pre-treatment is 4, as stated earlier in section 2.3 Ozonation and Flocculation Processes. The following figures 6-9 show the declining lines of all parameter measured during the experiment.
The series of data and curves showed in Table 5 and Figure 6-9 explain that all values i.e. conductivity, TSS, Pt/Co and COD, are declined. The experimental results show that TSS, Pt/Co and COD can be reduced in overall by about 99.83%, 99.99 and 37.54% respectively. Detailed removal efficiency of TSS, Pt/Co and COD are presented below.
Table 6. Removal Efficiency of TSS, Pt/Co and COD

| TMP   | Parameter          | Removal Efficiency (%) of TSS, COD and Pt/Co from: |
|-------|--------------------|-----------------------------------------------|
|       |                    | Raw to Ozonation                             |
| 3 bars| Ozonation to Membrane | 97.44, 98.08, 23.71                           |
| 5 bars| Ozonation to Membrane | 94.87, 98.93, 24.24                           |
| 7 bars| Ozonation to Membrane | 89.74, 98.08, 23.51                           |
| 9 bars| Ozonation to Membrane | 87.18, 98.08, 23.56                           |
| 11 bars| Ozonation to Membrane | 92.31, 98.08, 23.17                           |
|       | Raw to Membrane    | 99.89, 99.99, 38.03                           |
|       | Overall            | 92.31, 98.25, 23.64                           |

Raw to Ozonation means removal efficiency of TSS, Pt/Co and COD are 97.80, 99.19 and 18.20% respectively, from raw Batik wastewater to pre-treatment using flocculation and ozonation before filtered by membrane. Ozonation to Membrane with varied TMPs means removal efficiency of TSS, Pt/Co and COD from pre-treated Batik wastewater (Ozonation process) to membrane process. While Raw Membrane with varied TMPs means overall removal efficiency of TSS, Pt/Co and COD from the beginning (raw Batik wastewater) to the end of membrane process.

![Figure 10. Removal Efficiency from Raw Batik Wastewater to Ozonation Process](image)

It can be seen in figure 10, that both TSS and Pt/Co from Raw Batik Wastewater to Ozonation Process have significant removal efficiency, which are 97.8 and 99.19% respectively. On the contrary, removal efficiency of COD is only 18.20%.

Advanced Oxidation Processes (AOPs), specifically Ozonation, have been widely used for waste water treatment. AOPs have been developed to generate hydroxyl tree radicals through different methods,
which exposed the greatest ability to treat textile wastewater [19]. However, based on [19-23], there were no significant reduction in COD values, as well as occur in this research, only 18.20%, while removal efficiency of TSS and Pt/Co are significantly high.

The above comparison in Figure 11 show the removal efficiency of TSS between overall process since the beginning (raw Batik wastewater) until membrane process finished that show similar trends. From the figure, we can see that the highest removal efficiency of TSS values is at 3 bars for both condition, which gradually decrease at TMPs 5, 7 and 9 bars, but instead increase at TMP 11 bars. In terms of removal efficiency of TSS values, the best performance of polypropylene membrane is at 3 bars, followed by 5 bars and 7 bars, while the lowest performance is at 9 bars.

The above comparison in Figure 12 show the removal efficiency of Pt/Co between overall process since the beginning (raw Batik wastewater) until membrane process finished that show similar trends. From the figure, we can see that the highest removal efficiency of Pt/Co values is at 3 bars for both condition, which gradually decrease at TMPs 5, 7 and 9 bars, but instead increase at TMP 11 bars. In terms of removal efficiency of Pt/Co values, the best performance of polypropylene membrane is at 3 bars, followed by 5 bars and 7 bars, while the lowest performance is at 9 bars.

The above comparison in Figure 13 show the removal efficiency of COD between overall process since the beginning (raw Batik wastewater) until membrane process finished that show similar trends. From the figure, we can see that the removal efficiency of COD values is at 3 bars for both condition, which gradually decrease at TMPs 5, 7 and 9 bars, but instead increase at TMP 11 bars. In terms of removal efficiency of COD values, the best performance of polypropylene membrane is at 3 bars, followed by 5 bars and 7 bars, while the lowest performance is at 9 bars.
Similar to Figure 1, Figure 12 as well as Figure 13 show the similar trend for the comparison curve between the membrane and overall processes. Unlike the removal efficiency of TSS, which show the highest efficiency at 3 bars, both of Pt/Co and COD are have 5 bars for the highest removal efficiency, which are around 99.9% and 31.1% in average, respectively. In terms of removal efficiency of both Pt/Co and COD, the best performance of polypropylene membrane is at 5 bars, followed by 3 bars and 9 bars, while the lowest performance is at 11 bars.

4. Conclusion

In this study, the combination of ozonation (with flocculation) and membrane processes produced better results compared to separate or each process. For example, in terms of COD removal, the ozonation process gives a smaller efficiency, which is 18.20%, compared to the membrane process itself, which is between 37-38% depending on the TMP values used. However, the absent of pre-treatment with the Ozonation process may strongly lead to membrane fouling or even module damage.

The performance of an ultrafiltration polypropylene membrane during the treatment of Batik wastewater was evaluated by varying TMPs. The experimental results obtained that permeate flux increased linearly with the increase of TMP. In terms of removal efficiency, 2 of the 3 parameters (Pt/Co and COD) attained highest removal efficiency at 5 bars of TMP, while the other one (TSS) achieved at 3 bars of TMP.

The overall results of this study indicated that the combination of ozonation (with flocculation) and membrane processes was a promising method to treat Batik wastewater to adequately reduce TSS, Pt/Co and COD values. Therefore, the synergy of both methods is recommended to gain the removal efficiency. Furthermore, performance of the membrane could be improved by routine maintenance using suitable method [24, 25], thus could be used for longer time operation to treat more Batik wastewater and produce more clean water.

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