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HEAVY METAL CHARACTERISTICS OF WASTEWATER FROM BATIK INDUSTRY IN YOGYAKARTA AREA, INDONESIA

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ABSTRACT: Batik is Indonesian traditional textile product. The increasing use of synthetic dyes in batik production raises concern on heavy metal content in its wastewater. As wastewater from most factories is released to the environment without proper treatment, the pollutants including heavy metal content would pose health risks to humans and the environment. However, data on heavy metal characteristics of wastewater from batik industry in Indonesia is very limited. This paper presents the profile of heavy metal characteristic of 17 (seventeen) wastewater samples from batik factories in Yogyakarta Area, Indonesia. The total concentration of some heavy metals elements were measured by using inductively-coupled plasma mass spectrophotometry (ICP-MS). The result was compared to other studies and relevant local and international effluent standards. Hierarchical Cluster Analysis (HCA) was then performed to observe group of samples or parameters with similar characteristics that represent similar production method which affects their wastewater characteristics. The results showed that concentration of Cr, Co, Ni, Cu, As, Cd, and Pb ranged from 9.87 to 101 ppb, 0.03 to 3.89 ppb, 5.37 to 82.8 ppb, 19.7 to 472 ppb, 1.46 to 21.2 ppb, 0.03 to 20.3 ppb and 0.78 to 40.1 ppb respectively. In general, concentration of heavy metal in this study does not exceed regulation and also lower than result of other studies. HCA result confirmed that the source of Ni, Cr and Cu is from dye. HCA also indicated that other factors may affect the heavy metal concentration in batik wastewater which demands further study.

Keywords: Batik Industry, Wastewater, Heavy Metal, Cluster Analysis, Risk Assessment

1. INTRODUCTION

Batik is a traditional Indonesian textile product, although it is also developed in Malaysia [1]. Batik industry is an important economic sector in Indonesia. In 2019, export of batik products from Indonesia reached US$ 52.4 million. It is a significant number as compared to US$ 442 billion of textile product global market value of the same year [2].

Batik production method differs from common method used in textile industries. It involves the application of wax in its multiple coloring stages. A two colored batik sheet will require two times coloring stages. A three colored would require three times coloring stages, and so on. Part of the motive which is not intended to have the first color would be covered by wax to undergo the dyeing process of the first color. Afterward, the wax is removed and the process continues to the second coloring process by using the same method.

The application of wax, dye and also fixing agents in batik production results in wastewater characteristic of high pH, organic content (COD, BOD), total suspended solids (TSS) and color intensity [3–6]. Similar to the general textile wastewater, there is also concern on possible heavy metal content in batik wastewater [1,7,8]. Heavy metals are part of organic-based coloring dyes to enhance its absorption to fabrics or at least present as impurities of dyes [7,9]. Various heavy metal including Cd, Pb, Fe, Cu, Zn, Al, Mn, Mg, Ca, Cr and Si were detected in batik wastewater sample from Batik factory in Malaysia with similar production method as that is used in Indonesia [1,7]. The concentration for each element of heavy metal varies according to production stage[1].

In Indonesia, batik is produced mostly by middle to small scale industries which are categorized as home industry. One characteristic of home industries is their limited capacity in various aspects including occupational and environment health management. Factory workers are being exposed to chemicals involved in the production process with minimum personal protection equipment. On the other hand, batik wastewater from most factories are released to environment without proper treatment. While many batik factories are located in vicinity to housing areas, this improperly handling of wastewater may release pollutants to soil and groundwater. A study conducted by Syuhadah and Rohasliney (2015) showed a strong positive correlation between concentration of heavy metals measured in soil and plants surrounding a batik factory and heavy metal concentration in effluents from that particular batik factory [7]. This would pose health risk to human from direct use and exposure to contaminated soil, water bodies and groundwater. To develop appropriate measure to control this negative impact, risk based approach through risk assessment can be applied. Risk assessment consists of four generic steps, namely, (i) hazard identification, (ii) hazard assessment, (iii) exposure assessment, and (iv) risk characterization [10]. In the case of batik industry, hazard identification as the first step of risk assessment can be conducted through characterization of the source of hazard. In this case, it is wastewater. However, data on heavy metal characteristic of wastewater from batik industry in Indonesia is limited mostly to concentration of total Chromium (Cr), as it is the only heavy metal parameter regulated by Indonesia national standard [11]. On the other hand, study on batik wastewater are mostly focused on the development of treatment technology [12–14]. No study had been conducted to assess the risk can be posed by present wastewater handling to the environment and human health. This paper presents the profile of heavy metal characteristic of wastewater from batik factories in Yogyakarta Area as the initial phase of the risk assessment of batik industry in this area. The profile involves comparison of heavy metal concentration in this study to other studies, as well as to relevant effluent standards. Grouping of samples and parameters to examine factors affecting wastewater characteristic was conducted by using Hierarchical Cluster Analysis (HCA).

2. MATERIALS AND METHODS

2.1 Study Sites and Sampling

Special Region of Yogyakarta is one of provincial administrative area of Indonesia. It is located in Java Island, bordered by the Indian Ocean in the south and sharing land border with Central Java Province. It consists of four regencies, namely: Bantul, Sleman, Kulonprogo and Gunungkidul, and one city, Yogyakarta City as the capital (Fig.1).

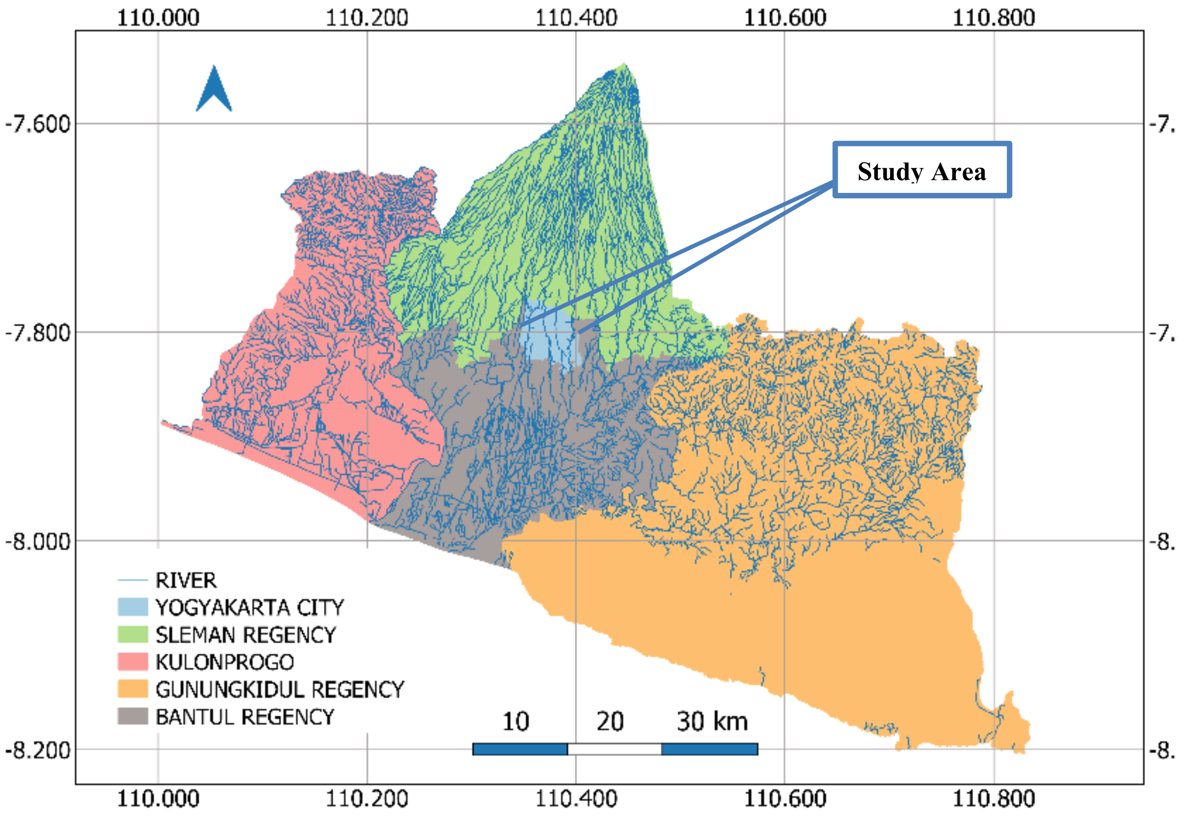

Fig. 1 Special Region of Yogyakarta and study area

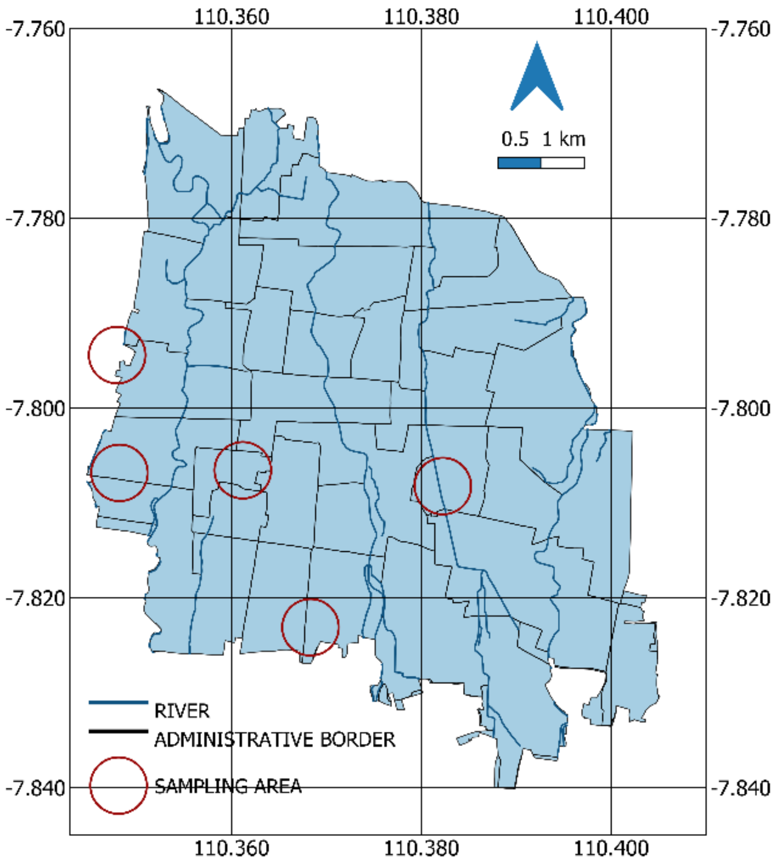

Fig. 2 Sampling area

Wastewater samples for this study were taken from batik factories located in Yogyakarta City and Bantul Regency (Fig.2). Samples were collected in polypropylene (PP) bottles. Parameter pH was measured on site by using Multiparameter Water Quality Checker Horiba U-50. Samples for heavy metal analysis were acidified with concentrated HNO_3 (grade for heavy metal measurement, 65%, Merck) until pH<2 for preservation. All samples were kept in 4° C until analysis.

Samples were wastewater after dyeing (step 4) and wax removal process (step 5) of batik production as referred to Fig. 3. Table 1 presents from which production process by referring to Fig.3 and type of dye used in the factories where the samples were taken from.

2.2 Samples Analysis

Prior to analysis, samples were pretreated according to United States Environmental Protection Agency (USEPA) method 3015A for microwave assisted acid digestion with modification [15]. Acid mixture of HNO_3 (grade for heavy metal measurement, 61%, Wako Chemical) and HCl (35-37%, Wako Chemical) were added to samples with the ratio of 9:1 and digested in laboratory microwave oven (CEM type MARS 6 using Easy Prep Plus vessel type). Digestion was set to pressure of 800 psi and temperature of 200°C, held for 15 minutes with ramping time of 15 minutes. Digested samples were then filtered by using 0.45 µm cellulose acetate membrane filter.

Table 1 Source of samples

Sample Code	Source of Process	Type of dyes used by factory
X1	After step 4	Naphtol
X2	After step 5	Naphtol
X3	After step 4	Naphtol
X4	After step 5	Naphtol
X5	After step 4	Naphtol, Indigosol
X6	After step 4	Naphtol, Indigosol
X7	After step 4	Naphtol, Indigosol
X8	After step 4	Naphtol, Indigosol
X9	After step 4	Naphtol, Indigosol
X10	After step 5	Naphtol, Indigosol
X11	After step 5	Naphtol, Indigosol
X12	After step 4	Remasol
X13	After step 4	Naphtol, Indigosol
X14	After step 4	Naphtol, Indigosol
X15	After step 5	Naphtol, Indigosol
X16	After step 5	Naphtol, Indigosol
X17	After step 4	Naphtol, Indigosol

Heavy metal content in samples was analyzed by Inductively Coupled Plasma Mass Spectrometry (ICP-MS) following USEPA method 200.8 [16]. Prior to measurement by ICP, digested filtered samples were diluted with 1 N HNO_3 solution. Standard solutions were prepared by dilution of standard stock (Wako Chemical) of each elements with 1 N HNO_3 solution. Isotopes used for measurement were ^{52}Cr, ^{59}Co, ^{60}Ni, ^{63}Cu, ^{75}As, ^{111}Cd and ^{208}Pb. For quality assurance of the measurement, internal standard ^{89}Y and ^{115}In were used to ensure stability during measurement. Limit of detection (LoD) for Cr, Co, Ni, Cu, As, Cd, and Pb were 0.18, 0.05, 0.31, 0.82, 0.07, 0.05, and 0.06 ppb respectively.

Heavy metal in textile related wastewater is associated with dyes which generally are organic compound [7]. Therefore, in addition to heavy metal, organic content in samples was also measured through the analysis of Total Organic Carbon (TOC). TOC measurement was conducted by using TOC analyzer type TOC-V CSN by Shimadzu following Standard Methods 5310B [17]. Samples were diluted and filtered to remove particulate prior to measurement. Standard solution for TOC measurement was prepared by using Potassium Hydrogen Phthalate or KHP ($C_8H_5KO_4$, 99.8-100.2%, Wako Chemical) powder which was dried and then diluted with milli-Q water to prepare necessary concentration of standard solutions.

2.3 Statistical Analysis and Map Works

Statistical analysis was conducted by using R software version 4.0.0 [18]. Basic R package "graphics" was used to present descriptive analysis result with boxplot graph. By using Mardia Test in "MVN" Package [19], it was checked that the data was multivariate normal after logarithmic transformation.

Finally, Hierarchical Cluster Analysis (HCA) was performed to group the samples by using Ward's linkage method [18]. To ensure comparability among datasets, standardization was performed before clustering. Dendrogram visualization of HCA was performed by using "factoextra" Package [20].

Map works in Fig. 1 and Fig. 2 was produced with QGIS open source software version 3.16 Hannover by using maps provided by Indonesia Geospatial Portal [21].

3. RESULTS AND DISCUSSIONS

3.1 Batik Production Process

In general, there are 2 types of batik which are differentiated by the device used to apply wax on the cloth. The first batik type is called '*batik tulis*" or hand painted batik in which a pen like device called "*canting*" is used during wax application. Another type is called "*batik cap*" or stamping batik in which a big stamp like device called "*cap*" is used during wax application. *Cap* is made of copper with usually a 20 x 20 cm^2 size. Basically, both types are produced through the process described in Fig.3.

1. Cloth is soaked in water for softening

2. Cloth is soaked in boiling water and then washed with detergent and dried

3. Batik craftsman will handpaint the motif by using wax to part of motif which will be left uncolored

4. Coloring process by soaking the wax painted cloth in the first color. Fixation agent and other chemical are also used to enhance the color intensity

5. Wax removal process by heating up the wax using hot water and then scrapping or sponging off the remaining wax afterwards

Fig. 3 Batik production process

Step 3-5 will be repeated according to the number of colour would be applied on each production. A "*canting*" is used in step 3 for hand painted type, while a "*cap*" is used in stamp batik making process. Chemical used during process consists of wax, colouring agent both natural or synthetic and fixation agent. Some most commonly used chemical are as presented in Table 2.

Table 2 Most common chemical used in batik industry [22]

Dye	Nat./Syn.*)	Fixation agent
Naphtol	Syn.	Diazonium salt
Remasol	Syn.	Water glass ($Na_2(SiO_2)_nO$)
Indigosol	Syn.	H_2SO_4, HCl
Indigofera	Nat.	Air
Teger, Jambal, Tingi, Maho-gany skin	Nat.	($K_2(SO_4)_2$), ($FeSO_4$), ($CaOH_2$)

*) Nat. = natural ; Syn. = synthetic

3.2 Laboratory Analysis Result

Total concentration of some heavy metal elements including Cr, Co, Ni, Cu, As, Cd, Pb as well as pH and Total Organic Carbon (TOC) are presented in Table 3 and boxplot presentation in Fig. 4, Fig.5 and Fig. 6.

Table 3 Descriptive statistics of laboratory analysis result

Parameter	Min.	Max.	Mean	SD
Cr (ppb)	9.87	101	36.9	25.0
Co (ppb)	0.03	3.89	2.05	1.14
Ni (ppb)	5.37	82.8	27.2	22.6
Cu (ppb)	19.7	472	120	110
As (ppb)	1.46	21.2	6.49	5.93
Cd (ppb)	0.03	20.3	3.79	5.34
Pb (ppb)	0.78	40.1	14.5	11.7
pH	6.21	11.1	8.83	1.62
TOC (g/L)	1.25	13.6	3.47	3.11

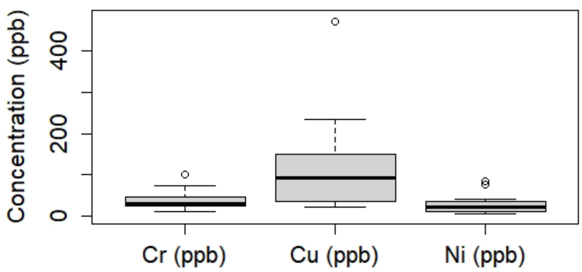

Fig. 4 Boxplot presentation of wastewater data for element Cr, Cu, Ni

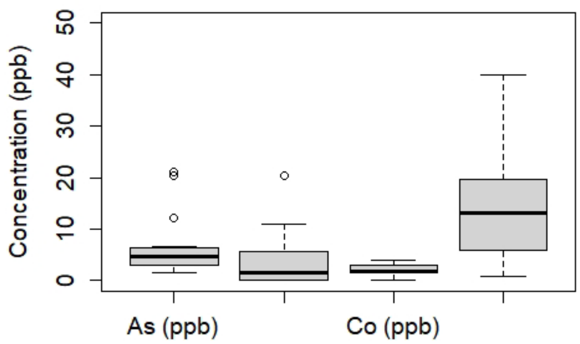

Fig. 5 Boxplot presentation of wastewater data for As, Cd, Co, Pb

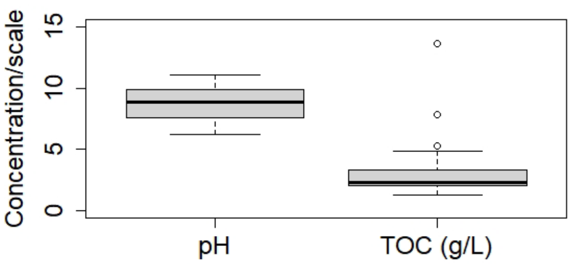

Fig. 6 Boxplot presentation of wastewater data for TOC and pH

Concentration were varied as showed in Table 3 and Fig. 4, Fig.5 and Fig. 6. Based on their standard deviation (SD), Cu had the highest variation among all measured metal elements followed by Cr, Ni, Pb, As, Cd and Co respectively. The same order was also applied to the distribution of heavy metal among samples which is Cu>Cr>Ni>Pb>As>Cd>Co. Cu had given the highest concentration compared to other elements. Studies conducted by [1] and [7] presents data on heavy metal concentration in batik wastewater following distribution order of Cu>Cr>Pb and Cu>Pb>Cd>Cr respectively. Compared with these 2 other studies, data in this study is consistent as Cu gives the highest concentration. Cu along with Cr, Ni and Co are predominant element for metal complex azo dyes [23] [24]. Included in azo dyes is naphtol which is commonly used in batik industry.

Heavy metal parameter which is being regulated in Minister of Environment Regulation No.5/2014 on textile wastewater effluent standard and Local Regulation of Yogyakarta Special Region No.7/2016 on Effluent standard for batik industry in Yogyakarta Special Region is only Total Chromium (Cr).

The maximum Cr concentration in batik effluent according to both regulations is 1 ppm. Compared to both standards, no sample exceed Cr maximum concentration. Compared to effluent standard in other countries such as Japan and Malaysia, concentration of heavy metal in all samples are also still below standard. Table 4 presents comparison of wastewater data to these standards.

As only Cr which is regulated for batik effluent standard in Indonesia, data on heavy metal content in batik wastewater in Indonesia was mostly limited to this parameter as reported by [11] and [25]. These studies reported the Cr concentration in batik wastewater samples taken from factories in Banyumas, Central Java Region and Sidoarjo, East Java Region respectively. Putra, Annisa and Budiarjo [12] reported concentration of Cr^{6+} and Pb in batik wastewater taken from one factory in Yogyakarta, the same area with this study. Data on other heavy metal elements in batik wastewater was from batik factories in Malaysia as reported by [1] and [7]. These studies reported heavy metal concentration in, samples taken from batik factories in Kelantan, Malaysia. Other heavy metal elements of Fe, Zn, Al, Mn, Mg, Ca, and Si with concentration of 3.0, 0.29, 12, 0.02, 0.97, 4.23, and 8965 ppm respectively were also reported in [1]. Compared to other studies, heavy metal concentration in samples in this study is relatively lower.

Table 4 Comparison of maximum concentration in samples (ppm) to effluent standards of Indonesia, Japan and Malaysia and to other studies

	Cr	Cr^{6+}	Cr^{3+}	Cu	Ni	As	Cd	Co	Pb
Effluent Standard									
A. Indonesia									
Local [1)]	1.0	-	-	-	-	-	-	-	-
National [2)]	1.0	-	-	-	-	-	-	-	-
B. Japan									
B [1)]	-	0.5	-	-	-	0.1	0.03	-	0.1
B [2)]	2	-	-	3	-	-	-	-	-
C. Malaysia									
C [1)]	-	0.05	0.20	0.20	0.20	0.05	0.01	-	0.10
C [2)]	-	0.05	1.0	1.0	1.0	0.10	0.02	-	0.5
Max. conc. in sample	0.01	-	-	0.47	0.08	0.02	0.02	0.00	0.04
Dewi, et.al [11]	1.1	-	-	-	-	-	-	-	-
Syahputra, et.al [12]	-	0.16	-	-	-	-	-	-	0.47
Suprihatin, et.al [25]	0.06	-	-	-	-	-	-	-	-
Moradi, et.al. [1]	0.08	-	-	0.29	-	-	-	-	0.04
Syuhadah, et.al. [7]									
Site A	0.00	-	-	0.19	-	-	0.01	-	0.16
Site B	0.00	-	-	0.57	-	-	0.01	-	0.34
Site C	0.00	-	-	0.02	-	-	0.00	-	0.47

A 1) Indonesian Minister of Environment Regulation No.5/2014
A 2) Local Regulation of Yogyakarta Special Region No.7/2016
B 1) Japan Standard for the protection of human health
B 2) Japan Standard for the protection of living environment
C 1) Malaysia Standard A: if discharged to inland waters within catchment areas
C 2) Malaysia Standard B: if discharged into other inlands waters or Malaysian waters

3.3 Hierarchical Cluster Analysis

Hierarchical Cluster Analysis (HCA) was performed to classify samples and parameters into different clusters. Two dendrograms were produced showing clusters of parameters and samples as presented in Fig. 7. and Fig.8.

Parameter cluster dendrogram shows that Ni and Cr belongs to the same cluster with the lowest dissimilarity. These parameters also in the same cluster with Cu with higher dissimilarity. This may indicate the Ni, Cr and Cu content in effluent were from the same source. Cr, Cu, Ni along with Co are said to be predominant element for metal complex azo dyes [23] [24] which are the type of dyes mostly used in batik production. Similar to textile wastewater, other source of heavy metals in batik wastewater can be from impurities of chemical used in different stages of production processes [26]. This is represented in dendrogram that other heavy metal elements belong to different cluster than Cu, Ni and Cr.

On the other hand, the sample cluster dendrogram shows that sample X17 and X4 are the most similar. However, sample X17 and X4 came from different production stage and X17 also used indigosol dye instead of only napthol like that of X4. This indicates factors other than the type of dye and production stage that affect wastewater characteristic. Also similar to textile wastewater, composition of batik wastewater may vary among factories due to variation of type of fabric, chemicals other than dyes, quality of water and equipment used for production [26].

4. CONCLUSIONS

Analysis on 17 (seventeen) wastewater samples from batik factories in Yogyakarta Region showed variation among elements and samples. The concentration of Cr, Co, Ni, Cu, As, Cd, and Pb ranged from 9.87 to 101 ppb, 0.03 to 3.89 ppb, 5.37 to 82.8 ppb, 19.7 to 472 ppb, 1.46 to 21.2 ppb, 0.03 to 20.3 ppb and 0.78 to 40.1 ppb respectively. In general, concentration of heavy metal in this study does not exceed regulation and also lower than result of other studies.

Hierarchical Cluster Analysis (HCA) produced dendrograms showing cluster of strong similarity among parameters Ni, Cr and Cu which indicates that the source of these metals are from dyes. HCA also indicates that factors other than the type of dye and production stage may affect heavy metal content in batik wastewater which should be clarified through further study.

5. ACKNOWLEDGMENTS

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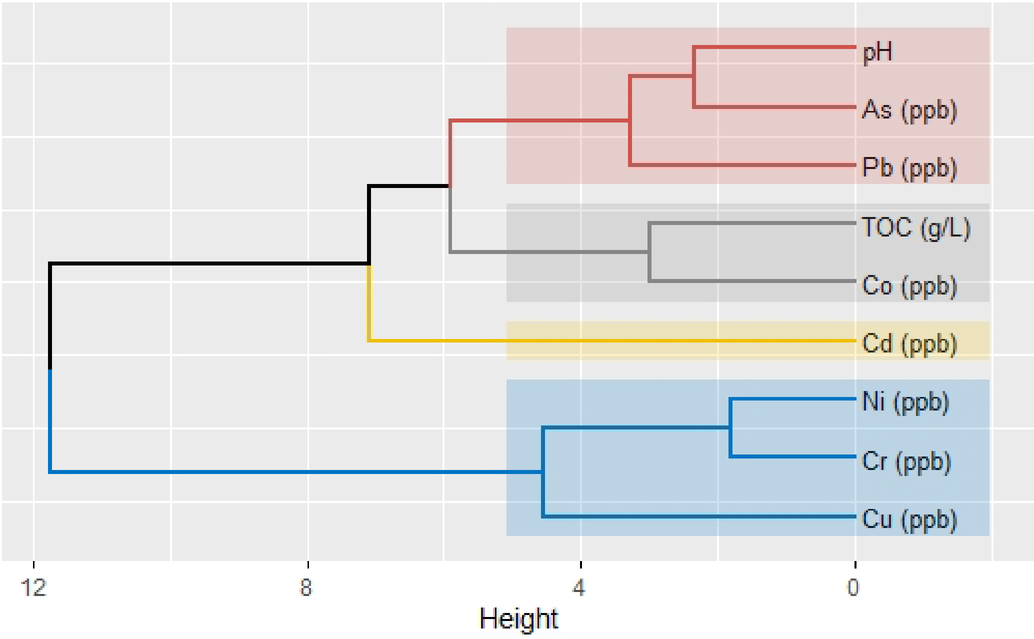

Fig.7 Dendrogram of Hierarchical Cluster Analysis (HCA) among parameters

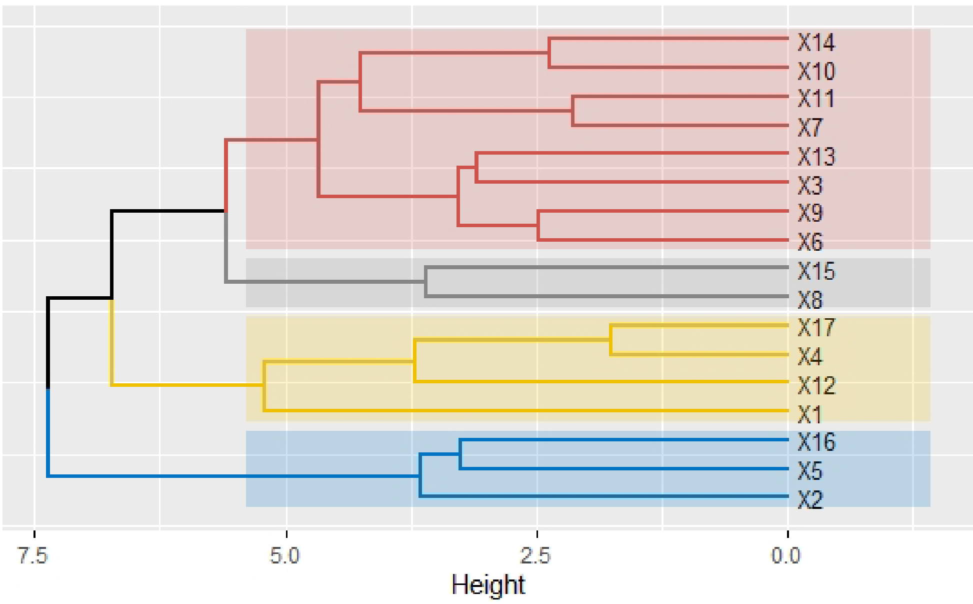

Fig. 8 Dendrogram of Hierarchical Cluster Analysis (HCA) samples

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