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Research article

An efficient and economical treatment for batik textile wastewater containing high levels of silicate and organic pollutants using a sequential process of acidification, magnesium oxide, and palm shell-based activated carbon application

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ABSTRACT

Considering the chemical properties of batik effluents, an efficient and economical treatment process was established to treat batik wastewater containing not only high levels of Si and chemical oxygen demand (COD), but also toxic heavy metals. After mixing the effluents obtained from the boiling and soaking steps in the batik process, acidification using concentrated hydrochloric acid (conc. HCl) was conducted to polymerize the silicate under acidic conditions. Consequently, sludge was produced and floated. XRD and FT-IR analyses showed that wax molecules were coordinated by hydrogen bonding with silica (SiO_2). The acidification process removed ~78–95% of COD and ~45–50% of Si, depending on the pH. In the next stage, magnesium oxide (MgO) was applied to remove heavy metals completely and almost 90% of the Si in the liquid phase. During this step, about 70% of COD was removed in the hydrogel that arose as a consequence of the crosslinking characteristics of the formed nano-composite, such as magnesium silicate or montmorillonite. The hydrogel was composed mainly of waxes with polymeric properties. Then, the remaining Si (~300 mg/L) in the wastewater combined with the effluents from the rinsing steps was further treated using 50 mg/L MgO. As a final step, palm-shell activated carbon (PSAC) was used to remove the remaining COD to < 50 mg/L at pH 3. Overall, the sequential process of acidification and MgO/PSAC application developed could serve as an economical and effective treatment option for treating heavily polluted batik effluents.

1. Introduction

The huge amount of waste originating from various manufacturing processes, such as the oil palm, paddy, rubber, cocoa, sugar cane, tea, and coconut industries, have jeopardized various environments in southeast Asian countries. Because these wastes are produced in industrial quantities, many studies have been conducted to reduce their adverse environmental effects.

While some traditional textile industries have been studied in terms of effluent quality, treatment, and hazardous effects (Kant, 2011), few studies have been performed on the overall status and treatment of batik wastewater. Most batik industries are relatively small family-based facilities that do not have wastewater treatment units. Due to their sporadic locations, it is difficult to establish centralized large-scale treatment systems.

Batik is one of the oldest cottage textile industries in Malaysia. Over 1000 batik factories are scattered mainly throughout Kelantan and Terengganu on the east coast of Malaysia (Rashidi et al., 2013; Redzuan and Aref, 2009). A major problem related to the batik industry is the discharge of wastewater produced during the soaking, boiling, and rinsing steps without proper treatment. This problem involves large volumes of water and chemicals such as waxes, dyes,

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and fixing agents like silicate, resulting in a high pH, chemical oxygen demand (COD_{Cr}), total suspended solids (TSS), and heavy metals (Arumai Dhas, 2008; Ghoreishi and Haghighi, 2003; Khalik et al., 2015). Because the presence of various chemicals in the effluents could threaten aquatic organisms due to their toxicities and non-biodegradability, it is more difficult to treat the water because of the complexity of the pollutants (Carmen and Daniela, 2012; Forgacs et al., 2004; Noor Syuhadah and Rohasliney, 2011; Rai et al., 2005). In the batik process, the three major sequential steps, soaking, boiling, and rinsing, which each have their own contaminants, lead to the complexity of wastewater treatment. In particular, wastewaters from the boiling and soaking steps have large amounts of COD, silicate, and heavy metals. Thus, an appropriate treatment is needed to remediate the effluents in compliance with local standards and regulations (Noor Syuhadah and Rohasliney, 2011; Saidah Malihah, 2010).

Currently, the major conventional methods for treating textile dye wastewater are physical and chemical treatments, because biological treatments are difficult to operate and usually require a long retention time, nor are they applicable for toxic heavy metal-containing wastewater (Aouni et al., 2009). Treatment using membrane filtration can reclaim water and chemicals, especially nano-filtration (NF) and reverse osmosis (RO); however, these are quite expensive and have significant fouling problems (i.e., RO) (Lau and Ismail, 2009). Moreover, ultrafiltration (UF) cannot be reused if the wastewater contains a high concentration of salt from the dyeing process (Babu et al., 1995). Electro-kinetic coagulation is feasible, but is also expensive in terms of energy costs and generates a high volume of sludge (Robinson et al., 2001). Activated carbon is economically attractive, but the removal efficiencies of dyes and other organic substances are fairly high only for a low range of concentrations (Carmen and Daniela, 2012). Photocatalysis have been studied on the remediation of textile wastewater (Cardoso et al., 2016; Soni et al., 2016; Souza et al., 2016), representing that organic dyes have been removed effectively.

Nevertheless, conventional technologies mentioned above could not be applied effectively to the batik wastewater that contains not only organic dyes, but also high contents of silica, wax and heavy metals. Accordingly, there is a continuous need for new technologies to treat batik wastewaters containing high levels of silicate, organic pollutants (i.e. wax), and heavy metals in an economical and effective manner.

In this study, for the first time, a sequential treatment method was developed through understanding the characteristics of batik wastewaters. The method developed consists primarily of three sequential steps. The first is to induce the polymerization of silicate via acidification using concentrated hydrochloric acid (conc. HCl). The resulting polymerized silica (SiO_2) could be coordinated with oxygenated groups of wax, the major organic pollutant in batik wastewater. In the second step, magnesium oxide (MgO) is applied to remove the remaining wax, silicate, and heavy metals. In the final step, palm shell-based activated carbon (PSAC) is used to remove the remaining organic fraction from the water. The main objectives of this study were to characterize actual batik effluents, to examine the feasibility of a sequential process of acidification and MgO/PSAC application for remediating wastewaters, and to determine the treatment mechanism.

2. Materials and methods

2.1. Materials

Actual batik wastewater was collected from the Dagang Batikraf factory located in Kelantan, Malaysia. The sequence of batik and wastewater production is shown in Fig. 1A. Wastewater samples were collected from four points in the batik process: soaking, boiling, and two rinsing steps. All collected samples were preserved at 4 °C prior to batch testing. Conc. HCl (37%, R&M) and powdered MgO (99%, R&M) were used for acidification and further water treatment. Granular-sized palm shell-based activated carbon (PSAC, 18 × 35 mesh-size, avg. size 0.707 mm) was obtained from Bravo Green Sdn Bhd, Kuching, Malaysia, and used as an absorbent to remove the remaining COD_{Cr}. The pH_{PZC} of PSAC was measured as described in the supporting information (SI).

2.2. Treatment process

Fig. 1B shows a schematic of the overall sequential treatment process. First, wastewaters obtained at the soaking and boiling steps in the batik process were mixed at a ratio of 70 mL boiling to 1000 mL soaking wastewaters. This ratio was the same as the production of wastewater on-site and elsewhere for batik production. Mixed wastewaters were kept for use in this research. After preparing mixed wastewater, conc. HCl was gradually added to adjust the solution pH to a predetermined pH (1, 2, or 3). During the addition of HCl, the mixture was stirred at ~30–50 rpm for approximately 15 min. Sludge was formed instantly, floating on the surface of the solution. The acidified suspension was kept calm until complete separation of the sludge from the liquid. The floating sludge was removed by filtration, and the remaining liquid phase (phase 1 liquid) was analyzed for heavy metals and COD, and then used for the next step. The sludge was also characterized using X-ray diffraction (XRD) and Fourier transform infrared spectroscopy (FTIR). Among the pH conditions tested, the optimum pH was chosen based on the removal of COD and heavy metals. The phase 1 liquid was further treated by the addition of different amounts of MgO (500, 750, 1000, or 1500 mg/L). The suspension was stirred at 50 rpm for 20 min and filtered to separate the liquid and solid (hydrogel) using 0.45 μm-pore filter paper prior to heavy metal and COD analyses. Because a hydrogel was produced during the application of MgO into the phase 1 liquid, the volumes were measured at the initial stage, after filtration, and after drying at 105 °C. The filtrate of the phase 1 liquid and two rinsing waters were mixed with a ratio of effluent rates and designated the phase 2 liquid that was further treated to remove the remaining Si with MgO at lower concentrations (20, 50, 75, or 100 mg/L). After adding MgO into the phase 2 liquid, the suspension was stirred for 20 min and filtered with 0.45 μm-pore filter paper. The filtrate was designated the phase 3 liquid and treated with PSAC to remove color and COD in the final stage. The phase 3 liquid was treated with different amounts of 18 × 35 mesh-sized PSAC (5, 10, 15, or 20 mg/L) for 8 h without pH adjustment or at pH 3. All operational temperatures were 27 ± 1 °C. Kinetic tests were also conducted using 15 g/L PSAC with an initial concentration of COD (322 mg/L) at 40 rpm for predetermined times (2, 4, 6, 8, and 10 h). Adsorption isotherms of COD were examined using the Langmuir and Freundlich models, while kinetic data were fitted with a pseudo second-order kinetic equation due to the chemisorption of organic dye on the pore surface of PSAC (Ho and McKay, 1998; Plazinski et al., 2013). All isotherms and kinetic models are described in the SI.

2.3. Analytical methods

Heavy metals in all samples were measured using inductively coupled plasma atomic emission spectroscopy (ICP-AES, Perkin Elmer model OPTIMA 8300). COD (potassium dichromate ($K_2Cr_2O_7$) method) was measured by the APHA method (5220 closed reflux, titrimetric standard method). XRD analysis (Empyrean Panalytical, the Netherlands) was used to characterize the qualification of inorganic matter in the sludges obtained at different pHs and the

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