



Coagulation–flocculation of azo dye Acid Orange 7 with green refined laterite soil



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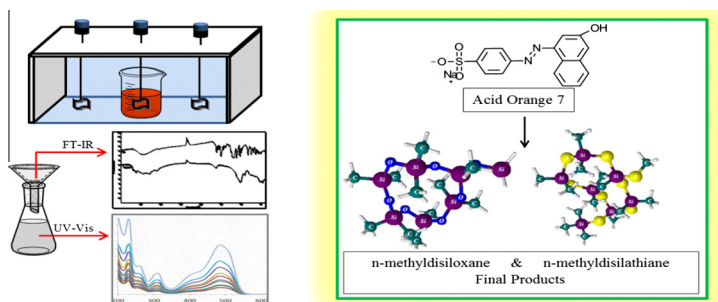
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HIGHLIGHTS

- Acid Orange 7 (AO 7) dye degradation pathway using laterite soil is established.
- Laterite soil is bi-functional (coagulation and flocculation).
- n-Methylsiloxane and n-methylsilathiane are formed via interparticle bridging.
- The present system controls release of sulfate ion to be exposed to environment.

GRAPHICAL ABSTRACT



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ABSTRACT

A novel degradation pathway of Acid Orange 7 (AO 7) by laterite soil in coagulation process is suggested. AO 7 has a strong azo bond links with benzene and naphthalene rings. The rings in AO 7 can be cleaved during coagulation and flocculation by laterite soil. Laterite soil rich in silica component could treat AO 7 with 98.43% color removal at pH 2. It could degrade azo dye molecule to the simplest hydrocarbon structure and non-toxic products in coagulation process through a linkage of reactions: C–N bond cleavage, desulfonation, silication, diazene reductase and polymerization. Ascertainment of the degradation pathway and products were characterized by UV–Vis and FT–IR techniques. The results show that laterite soil is bi-functional as a coagulant as well as a flocculant. Sludge Volume Index (SVI) with sludge generated as low as 21.703 mL/g provides evidence that, laterite soil has polymerization characteristic by forming n-methylsiloxane and n-methylsilathiane. Laterite soil detached sulfate ion in AO 7 to form n-methylsilathiane, indirectly preventing sulfate ion to be exposed to environment.

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1. Introduction

Degradation pathway of specific textile dyes has recently received attention from researchers. This happens because effluents processed from textile finishing companies have responded to the high mutagenic effects impacting the existing treatment plant

and water quality [1]. Degradation intermediates and byproducts may carry mutagenic and carcinogenic potential that would affect human health and environment [2]. Levin et al. [3] and Li et al. [4] reported that several intermediates and byproducts with toxic characteristics such as azo bond and aromatic amines have been released into water bodies. Azo dyes characterized by azo bond (–N=N–) structure responsible for the color richness have posed a major pollution problem to environment due to color visibility and toxicity. Azo dye molecules could not be broken down under

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Nomenclature

| | | | |
|-------------------------------|---|-------------------------------|-------------------|
| AO 7 | Acid Orange 7 | KBr | potassium bromide |
| ATR | Attenuated Total Reflection | mg/L | milligram/litre |
| Cl ⁻ | chloride ion | Na ⁺ | sodium ion |
| FT-IR | fourier transform infrared spectroscopy | NaCl | sodium chloride |
| H ⁺ | hydrogen ion | OH ⁻ | hydroxide ion |
| HCl | hydrochloric acid | S ²⁻ | sulfide |
| HN=NH | diazene | Si(OH) ₄ | silicic acid |
| HS ⁻ | sulfanide | SO ₃ ²⁻ | sulfite |
| HSO ₃ ⁻ | hydrosulfite | | |

the sunlight or radiant energy due to their high photolytic stable characteristic, thus critically affecting the aquatic system. During degradation, azo dyes cleavages potentially generate aromatic amines. Some aromatic amines are considered carcinogenic since it can accumulate in the food chain and imperil human health and ecosystem. Therefore, it is necessary to develop mechanism pathway of dye degradation to ensure that the degradation intermediates and byproducts yielded are safe to discharge into water streams.

A few methods have been proven to be effective for dye degradation [5–7]. To date, the pathway on the degradation of dyes in coagulation process is yet to be explored. Researchers have focused their studies on coagulation since it is a physicochemical treatment process that shows high performance in treating dyewastes [8,9]. Various inorganic coagulants such as aluminum, ferric, magnesium salts and lime had been applied alone to treat dye wastewater. However, the sludge produced from inorganic coagulant is toxic [10]. Flaten [11] reported that aluminum is a neurotoxicant product that contributes to Alzheimer's disease. Therefore, natural coagulants have recently received high degree of interest in treating dye wastewater.

In the present work, silicon (Si)-rich laterite soil as a natural coagulant was used in treating dyeing bath effluent. It was intended to investigate the degradation mechanism pathway of Acid Orange 7 (AO 7) using naturally prepared laterite soil. Observation of the AO 7 dye degradation under various pH and dosages of laterite soil coagulant was performed. UV–Vis spectra and FT-IR analysis were used to identify the degradation products during treatment and to establish a pathway of degradation. Degradation pathway can assist in ensuring zero potential hazards of intermediates and products formed. Determination of sludge generation in coagulation yielded from laterite soil was carried out by Sludge Volume Index (SVI) test. Besides, the wet sludge was tested for reusability.

2. Methodology

2.1. Dye

Acid Orange 7 (AO 7) with molecular formula C₁₆H₁₁N₂NaO₄S was supplied by Acros Organic. Stock solution of AO 7 was prepared by dissolving 1 g of AO 7 with deionized water in a 1 L volumetric flask. An appropriate amount of AO 7 from the stock solution was diluted to 500 mL with deionized water to prepare 50 mg/L of AO 7 sample.

2.2. Coagulant

The natural coagulant used in this study was laterite soil collected from Bukit Merah, Perlis, Malaysia. The characteristics of the laterite soil had been analyzed by using X-ray Fluorescence

(XRF) and the corresponding identified compounds are listed in Table 1. From the characteristic study, laterite soil shows the capability of the commonly utilized coagulants in treating wastewater. The reddish laterite soil was decolorized before being used as coagulant to treat synthetic dye wastewater. Flow chart for the decolorized method of reddish laterite soil is presented in Fig. 1. Initially, laterite soil was mixed with ultrapure water and blended, subsequently the mixture was filtered with a nylon cloth to screen out the large particles and impurities. The solution containing dissolved laterite soil was filtered through the nylon cloth. The laterite soil containing filtrate was collected and left to settle until double layers were observed. Since the hydrolyzed laterite soil has higher density compared to water, the upper layer was aqueous phase whereas the bottom layer was laterite soil paste. The aqueous phase was drained off and the laterite soil paste was dried under the sunlight. The procedure was repeated until the laterite soil was decolorized. The dried laterite soil was then crushed into powder and sieved through 75 µm sieve. The sieved laterite soil powder was mixed in ultrapure water and blended, followed by filtration to remove excessive fluid. Laterite soil paste collected on the filter paper was readily used as coagulant.

2.3. Coagulation experiments

Experiments on dyewaste coagulation were performed using Jar-Test apparatus (JLT6 VELD Scientifica). Jar test apparatus was used to determine the optimum pH. The initial pH of the solution was measured by 827 pH lab (Methrohm) and then adjusted to a range of values (pH 1, 2, 3, 5, 7, 9) using dilute HCl or NaOH solution (0.01–0.1 M). The mixtures of dye wastewater and laterite soil were stirred rapidly at 200 rpm for 2 min, then the stirring speed was decreased to 100 rpm for 15 min. Subsequently, the mixtures were allowed to settle for 30 min. 20 mL of the samples were collected from the top of the settling beaker (2 cm beneath the water surface). The collected samples were filtered through Whatman No. 4 filter papers of 12.5 cm diameter and 20–25 µm pore size. The filtrates were ready for analysis. The determined optimum pH was used and the subsequent experiments were continued by varying the dosage of laterite soil coagulant from 500 to 10,000 mg/L. The procedures were continued as mentioned above.

Table 1
Composition of laterite soil.

| Compound | Concentration (wt.%) |
|--------------------------------|----------------------|
| SiO ₂ | 36.3000 |
| Al ₂ O ₃ | 27.1000 |
| Fe ₂ O ₃ | 26.8600 |
| Others | 9.7400 |

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