REMOVAL OF REACTIVE BLACK 5 DYE USING POLY ACRYL AMIDE GRAFTED WATERMELON SEEDS IN SYNTHETIC DYE WASTEWATER

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To my beloved father, mother, husband and son.

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ABSTRACT

Reactive Black 5 dyes is one of the popular dyes that being used in dyes industry. Better shade, fastness and resistance towards organic solvent is one the main reason for the selection Reactive Black 5 dyes in dye industry. This properties of Reactive Black 5 dyes has lead to a several problem in the treatment process of the dye effluent. Besides that, presence of an aromatic rings in the structure of Reactive Black 5 dyes causes difficulties to remove the dyes stuff completely from the dye effluent. Hence, this study is focusing on the removal of Reactive Black 5 dyes particles by using grafted natural co-polymer from the grafting of Poly Acryl Amide with Watermelon seed (PAM-g-Watermelon seed).The synthesizing of PAM-g-Watermelon seed was done using microwave assisted method with ratio of 1:7 at irradiation time of 1 minutes where the grafting percentage was 189 %. Characterization of PAM-g-Watermelon seed before and after grafting was analyse using Scanning Electron Microscopy analysis (SEM) and Fourier Transform Infrared analysis (FTIR). Changes in morphological and addition of new band number that occur after grafting is the indication to prove that grafting process is successful. Performance of PAM-g-Watermelon seed was study by using Jar test method, where the dosage is varied at 0.2 g/L to 1.0 g/L, pH range at 3, 5, 7, 9, 11 and settling time was range from 60 minutes to 90 minutes with mixing speed of 140 rpm for rapid mixing and 40 rpm for slow mixing. For PAM-g-Watermelon seed best dosage was at 0.2 g/L with optimum pH 11 and best settling time range from 90 - 120 minutes.

ABSTRAK

Reaktif Hitam 5 pewarna adalah salah satu daripada pewarna yang popular yang digunakan dalam industri pewarna. Teduh, kelantangan dan ketahanan yang lebih baik terhadap pelarut organik adalah salah satu sebab utama pemilihan Reactive Black 5 pewarna dalam industri dye. Ciri-ciri pewarna Reaktif Hitam 5 ini menyebabkan beberapa masalah dalam proses rawatan efluen pewarna. Di samping itu, kehadiran cincin aromatik dalam struktur Reaktif Hitam 5 pewarna menyebabkan kesulitan untuk mengeluarkan bahan pewarna sepenuhnya dari efluen pewarna. Oleh itu, kajian ini memberi tumpuan kepada penyingkiran zarah-zarah pewarna Reaktif Hitam 5 dengan menggunakan polimer semulajadi yang dicelup dari percubaan Poly Acryl Amide dengan biji tembikai (PAM-g-Watermelon). Penyegerakan benih PAM-g-tembikai dilakukan menggunakan kaedah bantuan gelombang mikro dengan nisbah 1: 7 pada masa penyinaran 1 minit di mana peratusan cantuman adalah 189%. Pencirian PAM-g-Biji Tembikai sebelum dan selepas cantuman dianalisis menggunakan analisis Pengimbasan Mikroskopik Elektron (SEM) dan analisis Inframerah Transformasi Fourier (FTIR). Perubahan dalam morfologi dan penambahan bilangan band baru yang berlaku selepas cantuman adalah petunjuk untuk membuktikan bahawa proses cantuman berjaya. Prestasi PAM-g-Biji Tembikai dikaji dengan menggunakan kaedah ujian Jar, di mana dos berubah-ubah pada 0.2 g / L hingga 1.0 g / L, julat pH pada 3, 5, 7, 9, 11 dan masa penyelesaian adalah dari 60 minit hingga 90 minit dengan kelajuan pencampuran 140 rpm untuk pencampuran cepat dan 40 rpm untuk pencampuran perlahan. Untuk dos terbaik PAM-g-Watermelon ialah 0.2g / L dengan pH 11 optimum dan masa mendakan terbaik dari 90 - 120 minit.

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INTRODUCTION

1.1 Dyes Wastewater

A polluted natural water resource such as river, lakes, groundwater and etc has become a huge problem in this recent year. Contamination of these natural water resources was caused by many factors especially discharge from industry and sewage treatment plant. Of the contamination sources stated, dyes wastewater arise a major concern among the researcher to treat the polluted river water. Dyes wastewater also has a strong ability to pollute the water body either aesthetically, environment and human health. For instance, coloured river water will disturb the original view of the river due to presence of coloured river water. From environmental perspective, the concern was more focusing on the aquatic ecosystem and biodegradation process.

Dyes wastewater takes a long period to degrade due to the presence of aromatic compound in dye structure which provides resistance toward biodegradation process. In addition, classification of dyes were based on the application, chemical structure and a composed of chromophore that present in the structure of the dyes. Chromophore is the group of atom that responsible as a colour provider in the dye structure.

Chromophore containing centre were based on the types of functional group that attached on the chemical structure of the dyes; for example azo, anthraquinone, methane, nitro, arylmethane, carbonyls and etc (Christie, 2001). Furthermore, electron donating and electron withdrawing atom has intensify the hour of chromophore and hence often called as auxochromes. Major group of auxochrome were from amine, carboxyl, sulfonate and hydroxyl group (Dos Santos, 2007).

Azo dyes are an example of dyes that widely used in industrial processes. The effectiveness, fastness and cost effective of azo dyes were the main reason why azo dyes were preferred to be used in industrial processes. However, azo dyes have a carcinogenic and mutagenic effect. These carcinogenic and mutagenic effects may be due to the direct action of the agent itself or due to the generation of arylamine derivatives during the transformation of azo bond (Rajaguru, 1999).

Synthetics dyes residuals in wastewater come from various resources such as textile industry, pharmaceutical industry, tannery industry and many more. Out of the listed industry, textile industry has been labeled as the main industry that contributed to the highest synthetics dyes pollution. According to Baban *et.al* (2010), textile industry was estimated to produced 7 x 10^5 tonnes wastewater was per year and 10 to 25 percent of dyes was lost during dyeing process and 2 to 20 percent was directly discharged into waterways.

Treatment of dye containing wastewater involves a treatment of highly houred wastewater that contains various types of dyes with different concentration. These wastewater should be treated and comply with the discharged standard as stated in EQA 1974 under Regulation of Industrial Discharge 2009. Synthetics dyes are widely used in industry such as textile, paper, pharmaceutical, petroleum and photography (Sathian *et.al*, 2013, Marmion, 1991). Discharged of dye containing wastewater, could harm the environment as well as bring an adverse effect towards human health. This is because synthetics dyes contain aromatic compounds such as naphthalene which is known to be having toxicity, carcinogenic and mutagenic properties (Suteu and Zaharia, 2009).

Typically, dyes wastewater are the most problematic to be treated due to their chemical stability, high chemical oxygen demand (COD), toxicity effect and some of these dyes are suspected as carcinogenics (Syafalni *et.al*, 2012) . According to Fahmi (2010), the presence of synthetics dyes even in a little amount are easily seen by naked eyes if presence in water body. This small portion of synthetics dyes not only disturb the aesthetics value of water body but at the same time could affect the aquatic ecosystem by limiting the penetration of sunlight (Fahmi *et,al*, 2010).

According to Chequer *et.al*, (2013), there are 10,000 types of dyes and hour pigment were used in industrial process, and it is estimated that there are 7 x 10^5 tons of synthetics dyes were produced every year. Besides that, in textile industry it is estimated that almost 200,000 ton of dyes was lost to the effluent annually during dyeing and finishing process. This effluent will bring an adverse effect towards environment since dyes molecule was not easily degrade and has high stability towards light, temperature, chemicals, soap, water and detergent (Couto, 2009). Resistance towards biodegradation of dyes wastewater was caused by present of aromatic compound in dyes structure.

Furthermore, huge amount of water used by textile industry has generated huge amount of dyes wastewater which consider most polluted effluent with various harmful composition. Dyes wastewater possesses a fluctuation of Biochemical Oxygen Demand (BOD), Chemical Oxygen Demand (COD), pH, colour and salinity. The composition of dye wastewater were influence by the variability of organic-based compound used in the process and type of chemical and dyes used in the processing unit (Mansour *et.al*, 2012).

Previously, there are many researchers have summarized a number of studies on physicochemical method for the removal of dyes from wastewater such as coagulation and coagulation, advanced oxidation, activated carbon, ozonation and photocatalysis. Eventhough these methods are effective in dye removal but several drawbacks are either costly to apply in industries or produce secondary sludge, limits their application (Ventaka, *et.al*, 2005).

Hence, the releasing of dyes wastewater into environment endangers human health as well as environment since most treatment plant was less effective in the removal of hour and carcinogenic or mutagenic properties of dye compound. Difficulty to treat dyes wastewater.

1.2 Coagulation

Coagulation is one of the vital processes in wastewater treatment. This process is the process where the colloidal particles are destabilized. After that, the unstable colloidal particles will agglomerate to form larger flocs which then settle by gravity. Furthermore, this process will help to enhance the clarity of the treated discharge.

Besides that, there are various types of coagulants being used such as natural coagulants, synthetic coagulant and also the hybrid of naturals and synthetic coagulants. This coagulant was selected mainly based on the suitability of the wastewater constituent which related to the positive and negative charge present in the wastewater and also operational cost.

Moreover, synthetic coagulant can be divided into anionic, cationic and non-ionic polymer and usually has very high efficiency but it is costly besides has a potential hazard towards environment and human health (Droste, 1997).Common types of coagulants that usually used is aluminium sulfates and iron salts. Multivalent characteristic of these two cations effectively attracts colloidal particles and their relative insolubility enhanced their removal efficiency to a high degree.

In addition, treatment of industrial wastewater by using synthetic chemical as coagulant has been proven could bring an adverse effect towards environment and human health. According to Muhammad *et.al* (2015), residual of aluminium salts in treated wastewater has been proven to be one of the contributing factor to Alzheimer's disease.

Besides that, the used of aluminium salts in wastewater treatment can cause the excessive production of sludge (Muhammad *et.al,* 2015).Excessive sludge production required high sludge treatment cost in order to ensure the sludge was properly treated and less harmful towards environment. Furthermore, Ghebrimichael, (2004) stated that some synthetic monomers such as acrylamide show neurotoxicity and carcinogenic properties.

1.3 Problem Statement

Treatment of dyes wastewater is one of the issue that still unsolve until today. One of the dyes that became a concern is Reactive Black 5 dyes. Reactive Black 5 dyes is a common dye that used in dye industry. Better shade, fastness and resistance towards organic solvent is the reason why Reactive Black 5 is being choose. In addition, presence of an aromatic ring structure in Reactive Black 5 enhance the difficulty to treat the Reactive Black 5 dye completely from wastewater. Hence, to encounter this problem many research is done to develop a new potential coagulant derived from natural grafted polymer.

Furthermore, Chitosan-g-PAM and Pandan leaves Cellulose-g-PAM is the example of grafted natural co-polymer that being used to treat Reactive Black 5 dye (Norzita *et.al*, 2013 and Noor Yahida *et.al*, 2014). Previous study by Muhammad *et.al,* (2015) prove that watermelon seed having a potential as coagulant to treat raw water, but there is no research is done on the potential of watermelon seed grafted PAM in treating Reactive Black 5 dye.

Hence, this study will focused on the removal of Reactive Black 5 by using PAM-g-Watermelon seed in synthetic dye wastewater. PAM was selected in this study because it is non-toxic polymer with high molecular weight and positive charge. High molecular weight of PAM causes it able to extend it branches longer so that the availability of loop for contaminant to attached is more and positive charge of PAM could attract more Reactive Black 5 particle and will enhance the coagulation process.

1.4 Objectives

1) To synthesize and characterize the coagulant from grafted Poly Acryl Amide watermelon seed.
2) To study the effect of variable such as pH, settling time and dosage on the removal efficiency of grafted Poly Acryl Amide watermelon seed on the removal of Reactive Black 5 dyes from synthetics dyes wastewater.
3) To analyse the removal efficiency of grafted Poly Acryl Amide watermelon seed on the removal of Reactive Black 5 dyes from synthetics dyes wastewater base on COD, TSS, turbidity and colour reduction.

1.5 Scope of Study

1) To synthesize the coagulant from grafted Poly Acryl Amide watermelon seed by using the microwave assisted method and characterize of the watermelon seed was using Scanning Electron Microscopy for morphological and FTIR to determine the functional group presence in the grafted coagulants.

2) To study the performance of PAM-g-Watermelon seed at varies dosage (0.2 to 1.0 g/L), at pH 3, pH 5, pH 7 pH 9, pH 11 mixing speed at slow mixing was 40 rpm, while for rapid mixing was 140 rpm on the removal efficiency of grafted Poly Acryl Amide with watermelon seed in the removal Reactive Black 5 dyes from synthetic dyes wastewater.

3) To analyze the removal efficiency of grafted Poly Acryl Amide with watermelon seed in the removal of Reactive Black 5 dyes from synthetic dyes wastewater through the analysis on COD, colour, turbidity and TSS removal by using mathematical and statistical analysis (Anova).

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