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Green synthesis of silver nanoparticles doped activated carbon for Rhodamine B dye adsorption

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Abstract. Application of dye is one of the major industry with various application from everyday attire to food colouring. However, dye pollution is serious and threatening issue that need to be address to avoid harmful effect towards humans, animals and environments. In this study, silver nanoparticles (Ag NPs) were synthesised via *Clitoria Ternatea* (CT) flower extract and doped with activated carbon (Ag NPs/AC) to act as adsorbent in removing rhodamine B dye (RB). CT Flower mediated green synthesis for Ag NPs provide safe, low cost and sustainable method compared to conventional chemical and physical synthesis. UV-visible spectroscopy was used to analyse the green synthesis for the formation of Ag NPs with peak absorbance at 400 nm. The effect of activated carbon (AC) mass loading, contact time and adsorbent dosage were analysed to study the adsorption capacity of Ag NPs/AC with viable condition of 0.75 g AC mass and 30 mg dosage with complete removal at 90 minutes. Hence, incorporation of Ag NPs with AC contributes to better adsorption properties for the removal of RB dye.

1. Introduction

Clean water and sanitation in sustainable development goals (SDGs) specifically SDG 6 need to be actively promoted and support since the world population is increasing. Water as basic needs for all living things is a valuable and vital commodity for sustenance of life [1]. However, water pollution associated with toxic pollutants is a serious and significant problem majorly anticipated from industrialization [2]. Hence, there are needs in removing water pollutants which source from industry before being discharge to water sources. There has been a lot of concern towards organic pollutants such as dyes, pharmaceutical and agriculture waste as toxic water pollutants. Among them, dye pollutants are gaining attention due to its wide application in cosmetics, rubber, painting, leather and textile industries [3]. Toxicity of dye pollutants which affected aquatic life and humans are the major concern since more than 100,000 types of commercial dyes are expected to produce annually [4]. For instance, Rhodamine B dye (RB) which often used in industries such as food, paper and textile contribute to massive application [5]. Even so, skin irritation, eyes and respiratory damage are the effect of the exposition towards RB dye for humans and animals. Moreover, chronic poisoning, neurotoxic and carcinogenic are



the effect from RB dye pollution in drinking water [6]. Therefore, removal of RB dye is crucial for the development of a healthy and safe environment.

Various dye removal technologies had been applied which include biological, chemical and physical methods. Bacteria, yeast, algae and fungi that are used in biological method reduce harmful components in dye [7]. Chemical techniques in removing dye include ozonation, electrochemical and photocatalytic degradation. Moreover, physical method include adsorption, membrane filtration, precipitation-flocculation and ion exchange in removing dye pollutants [1]. However, major drawback of using chemical treatment is by-products produced that can lead to secondary pollution. Biological treatments are time consuming and not all physical treatments suitable for all dyes [8]. Therefore, there is a need to apply for a dye removal method that will not have any by-products, fast and compatible with various dyes. Among the technologies highlighted, adsorption is considered as promising method in removing dye pollutants. Adsorption is a separation process in which solid adsorbent attracted components in fluid phase to its surface and forming attachments to remove the fluid components [9]. Dye pollutant removal via adsorption process can provide high efficiency, simple operation, low cost and less chances to produce secondary pollutants [10, 11]. Hence, adsorption process in removing dye is by far the most efficient and extensively use for both organic and inorganic dye [12].

Silver nanoparticles doped activated carbon (Ag NPs/AC) is a functional material with various application mainly in adsorption and antibacterial activity. Previous work proven the effectiveness of Ag NPs/AC in removing dye and water pollutants such as phosphate [10], crystal violet [3, 12], malachite green [13, 14], methyl orange [15, 16] and methylene blue [14, 17] with no study in removing RB dye. Silver nanoparticles (Ag NPs) can enhance the active sites of activated carbon (AC) for better adsorption capacity. However, from the best of the author's knowledge, preparation of Ag NPs from these studies only include polymer based and chemical synthesis with no study using flower extract based synthesis of Ag NPs for the removal of RB dye. Furthermore, preparation of Ag NPs using flower extract is one of the techniques in green synthesis that can contribute to low energy used, renewable materials, minimal waste and risk methods [18]. *Clitoria Ternatea* (CT) flowers which abundantly available in tropical Asia [19] successfully assisted in the preparation of several nanoparticles (NPs) such as gold NPs [19] and Ag NPs [20, 21] which function as stabilizing and capping agent. In this study, green synthesis of Ag NPs via CT flowers extract (CTFE) and loaded into AC was prepared as solid adsorbent for RB dye removal with few parameters contributed to adsorption performance which include AC mass, dosage and contact time.

2. Materials and methods

All chemicals used are analytical grade. Fresh CT flowers were harvested from a farm in Kuala Lumpur. AC powder was purchased from Chemiz, Malaysia. Meanwhile, hydrochloric acid (HCl), silver nitrate (AgNO_3), RB dye and sodium hydroxide (NaOH) were purchased from R&M Chemicals, Malaysia.

2.1. CT flower extract preparation

The methods in this study follow our previous works Mohamad Aizad et al. [3] for the preparation of CTFE and adsorbent synthesis. Aqueous CTFE preparation started with cleaning and rinsing the fresh flowers with DI and tap water to remove sand, impurities and dirt. The cleaned flowers were dried at 70°C for 2 hr. Next, 100 ml of DI water were mixed with 50 mg of dried CT flowers powder at room temperature with continuous stirred for 1 hr. The aqueous extract was centrifuged for 30 min and filtered to remove all solid and flowers impurities. Finally, the prepared flower extract was kept in a freezer to remain its chemical structure.

2.2. Green synthesis of Ag NPs/AC

Both 3 mM of AgNO_3 and 0.1 M of NaOH were prepared beforehand for further use in the preparation of Ag NPs/AC. 4 ml of 3 mM AgNO_3 was mixed for 10 min with AC of 0.25 g, 0.5 g, 0.75 g, 1.0 g and 1.25 g and named as 0.25SAC, 0.5SAC, 0.75SAC, 1.0SAC and 1.25SAC respectively. Then, 20 ml of

prepared CT flower extract was mixed continuously for 10 min with 1 ml of 0.1 M NaOH. Next, the flower extract was transferred into the mixture of AgNO₃ and AC with continuous stirred at 50°C for 30 min. Subsequently, the mixture was centrifuged, washed and sonicated to ensure high purity of Ag NPs/AC to be collected. Finally, the mixture was dried for 1 day and ready to be collect. Figure 1 shows the overview of the whole process starting from Ag NPs/AC green synthesis until adsorption process.

2.3. Characterization

Formation of Ag NPs using green synthesis was characterized via UV-Visible Spectroscopy (UV-Vis) (Shimadzu UV-2600) to identify and study the peak absorbance that range from 200 – 700 nm.

2.4. RB dye adsorption

10 mg/L of RB dye was prepared for adsorption process. Orbital shaker (IKA Shakers KS 260 basic) was used in the process of adsorption with constant 300 rpm. The initial concentration of RB dye solution and mixture of Ag NPs/AC with RB dye were collected to be analysed at certain time interval using UV-Vis to study the adsorption capacity. 10 mg of 0.25SAC, 0.5SAC, 0.75SAC, 1.0SAC 1.25SAC and pure AC (PAC) were mixed with 50 ml of 10 mg/L RB dye individually as initial assumption to study the effect of AC mass and contact time. The percentage of RB dye adsorption can be calculated using equation 1 below extracted from UV-Vis data where abs is absorbance.

$$\text{RB dye adsorption \%} = \frac{\text{Initial abs} - \text{Final abs}}{\text{Initial abs}} \times 100\% \quad (1)$$

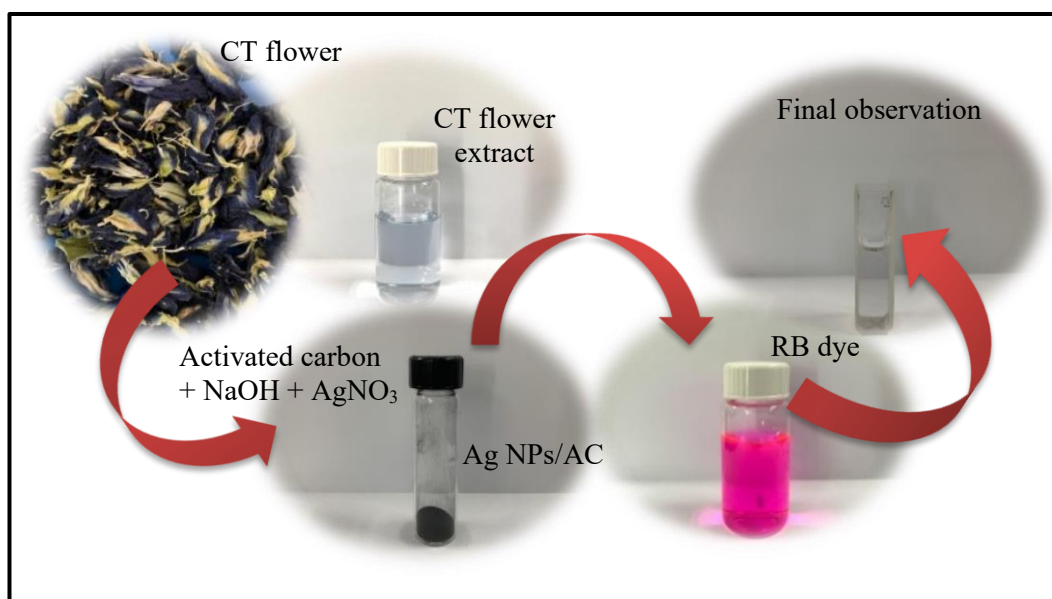


Figure 1. Overview of RB dye adsorption procedure using Ag NPs/AC as adsorbent

3. Results and discussions

3.1. Green synthesis of Ag NPs/AC

Preparation of Ag NPs via CTFE was successfully synthesized in this study. Green synthesis using flower extract provide fabrication for different shapes and sizes and considered as simple method [22]. Naked eye observation shows colour shifting from light blue (CTFE) to dark brown. Moreover, the green synthesis of Ag NPs using CTFE was illustrate as shown in Figure 2 which indicates the proposed formation of Ag NPs with CTFE that function as capping and reducing components from Ag^+ to Ag^0 . The usage of CTFE will replace the conventional sodium citrate, sodium borohydride and hydrazine which used in chemical synthesis of Ag NPs. Meanwhile, the synthesis of Ag NPs/AC was rapid with major black and small amount of silver powdered compound were produce with different loading mass of AC. Deposition of Ag NPs into AC provide higher reactive center and adsorption capacity as the Ag NPs is in nanosize and higher surface area [12].

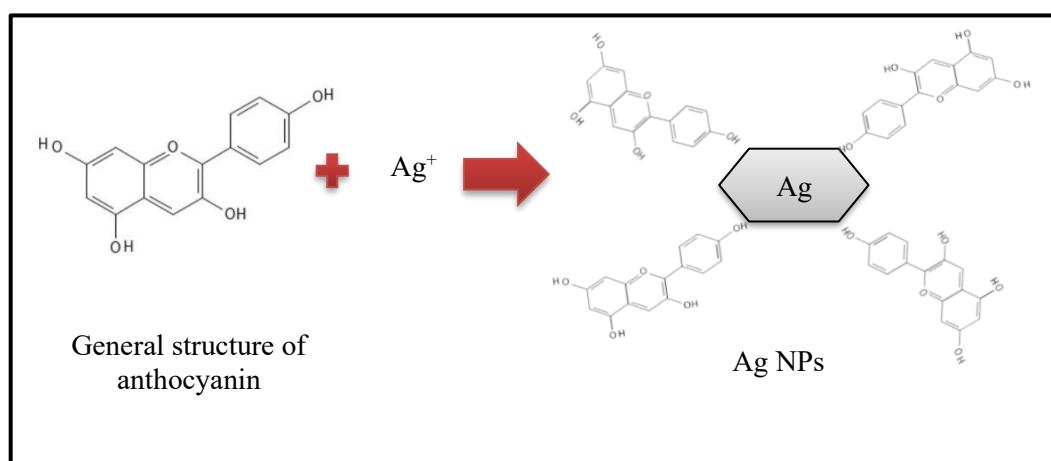


Figure 2. Proposed schematic presentation of Ag NPs formation with anthocyanin.

3.1.1. UV-Visible Spectroscopy analysis. The formation of Ag NPs was analysed using UV-Vis with different time interval to study the time taken for a complete reaction and peak shifting. Figure 3a shows the CTFE spectrum with two visible peak at 250-300 nm and 550-600 nm which indicates the presence of anthocyanin and flavonoid components [23] which are the vital elements in green synthesis of Ag NPs. Figure 3b, 3c and 3d shows peaks at 400-407 nm which is the normal range peak for Ag NPs. Peak formation of Ag NPs at 10 min was 407 nm and shift to lower wavelength at 400 nm for 30 min indicates the synthesised Ag NPs was from larger to smaller nanoparticle. This is contradicted to works from Khwannimit D et al. (2020) [21] that produce Ag NPs at 430 nm which considerably bigger size compared to this study. However, since this study particularly applied Ag NPs and doped with AC for adsorption application, smaller size of Ag NPs provides good benefit as it is high surface area.

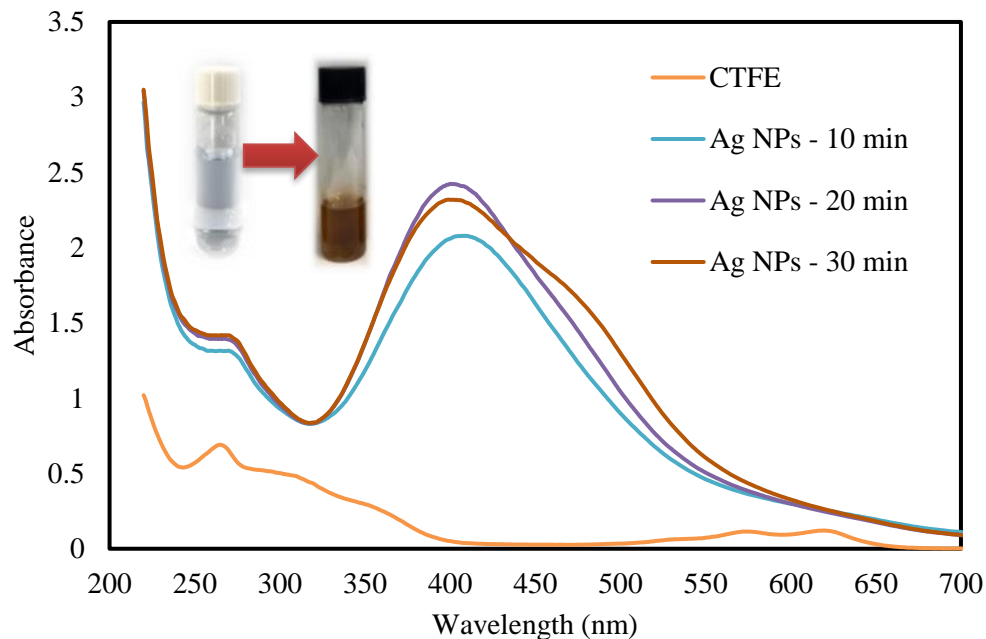


Figure 3. UV-Vis spectra of a) CTFE b) Ag NPs – 10 min c) Ag NPs – 20 min d) Ag NPs – 30 min

3.2. Batch RB dye adsorption

3.2.1. Effect of activated carbon mass. The adsorption capacity for 0.25SAC, 0.5SAC, 0.75SAC, 1.0SAC, 1.25SAC and PAC were tested with dosage of 10 mg to study the effect of ratio or mass variation of AC in Ag NPs for adsorbing RB dye. Figure 4a shows that 0.75SAC had better adsorption capacity with 19% removal compare to other ratio and PAC itself. This is because Ag NPs can enhance the active site of AC and increasing the surface area [24] to adsorb more RB dye compare to PAC. However, other ratio did not perform well maybe due to the unsuitability of the combination of both Ag NPs and AC. Hence, 0.75SAC shows better immobilization of Ag NPs and AC in removing RB dye.

3.2.2. Effect of contact time. The adsorption of RB dye took more than 240 min to removed 25% RB dye as shown in figure 4b where 0.75SAC with 10 mg dosage were used in this analysis. The adsorption trend shows positive output in removal of RB dye and it is expected to increase with time. But it is reported that at certain time the adsorption will constant as the active site decreases [12]. However, due to less amount of dosage used in this study, the time taken in removal of RB dye takes longer time. Hence, for 10 mg dosage it requires more than 240 min to remove RB dye.

3.2.3. Effect of dosage. 10, 20, 30 and 40 mg of 0.75SAC were tested to adsorb RB dye to study the effect of dosage. Figure 4c shows that increasing in dosage can increase the adsorption of RB dye due to large availability of active site in AC [12]. 30 and 40 mg shows better adsorption since it only takes 30 min to remove RB dye around 73% and 87% respectively. However, 30 mg was choosing as the best dosage because there is no significant difference compare to 40 mg. This is to ensure less amount of dosage used and to avoid secondary pollution.

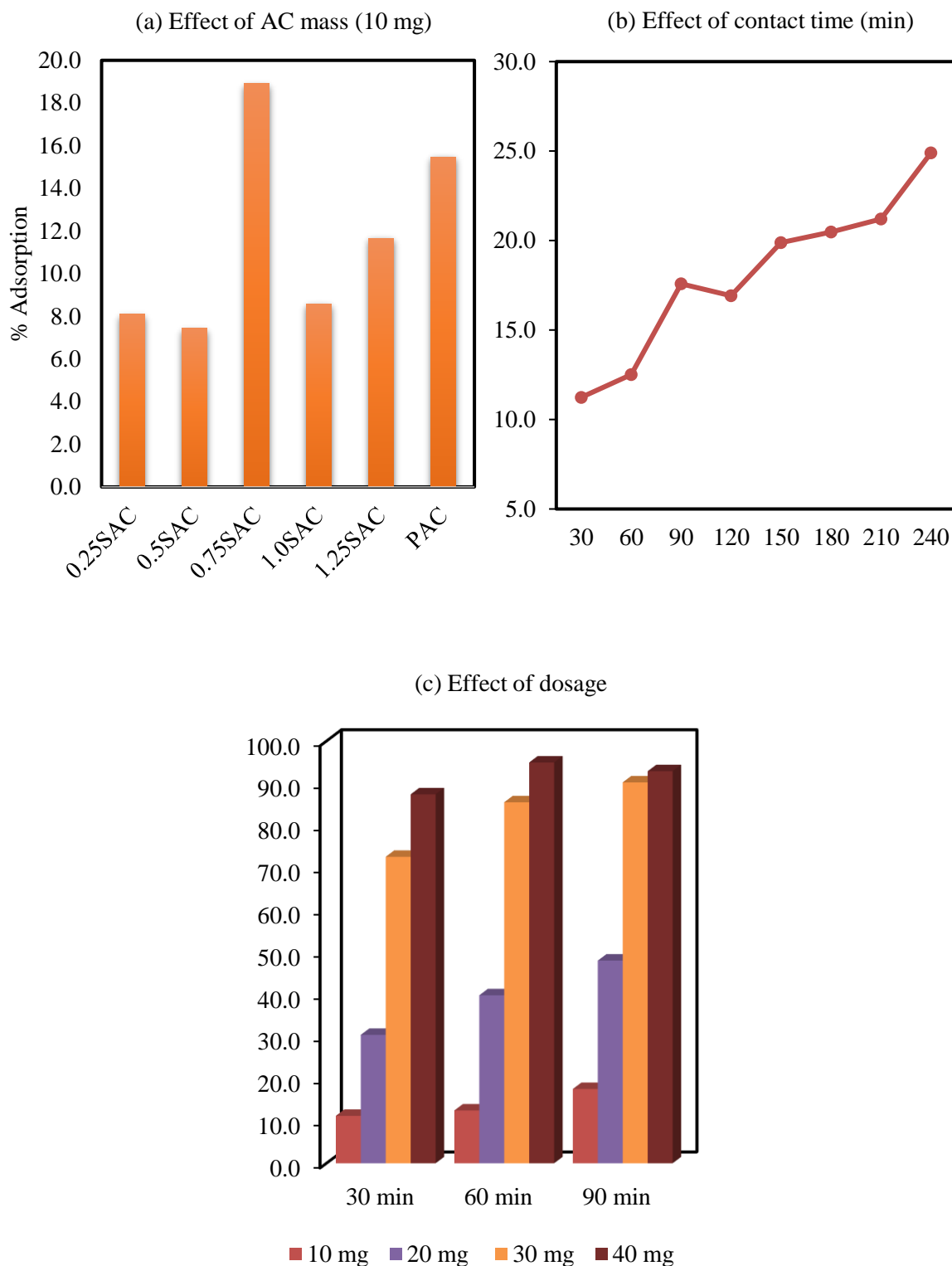


Figure 4. Adsorption capacity study for (a) AC mass (b) contact time and (c) dosage [Speed: 300 rpm, Temperature: 26°C, pH: 7]

4. Conclusion

The green synthesis of Ag NPs/AC was successfully carried out using CTFE and doped with AC as functional material for adsorption capacity study. It is known that the synthesis method was rapid and small Ag NPs produced with absorbance peak at 400 nm. Furthermore, the adsorption capacity study shows that Ag NPs/AC are capable for adsorbing RB dye. Ag NPs/AC with AC mass of 0.75 g shows better adsorption compared to PAC indicating suitable doping between amount of Ag NPs and AC. The effect of contact time reveal that it needs more than 240 min for 10 mg dosage to completely adsorb RB dye while 30 mg and 40 mg dosage can adsorb above 80% for the first 60 min indicating high availability of active sites that were enhance by Ag NPs. Hence, application of Ag NPs/AC as adsorbent to remove RB dye was successfully conducted with viable condition and potentially applied in the wastewater treatment industry.

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