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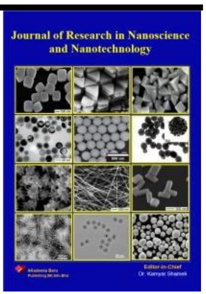

# Silver Nanoparticles Loaded Activated Carbon Synthesis Using *Clitorea Ternatea* Extract for Crystal Violet Dye Removal

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## ABSTRACT

Dyes are coloured compound which are widely used in textile, painting, rubber, cosmetics, plastics and leather industry to colour their products. However, the irresponsibility of certain manufacturer results in producing dye waste and channel it to water resources had become one of the biggest challenges in water pollution. In this study, an effective solid adsorbent derived from sustainable sources for adsorption capacity study was produced which is silver nanoparticles loaded activated carbon (Ag NPs – AC) to remove crystal violet (CV) dye. Adsorption process are cost – effective, simple and flexible with various dye pollutants. Silver nanoparticles (Ag NPs) was synthesized from *Clitorea Ternatea* flower extract that utilizes functions as stabilizing agents for silver nitrate ($AgNO_3$) to promotes environmentally friendly with no toxic chemicals produced and loaded in activated carbon (AC). Characterization of Ag NPs was analysed using UV-Visible which correspond to peak at 408 nm and XRD analysis. Four peaks values for silver at $2\theta$ of 38.19°, 44.43°, 64.57°, 77.43° and average crystallite size of Ag NPs and Ag NPs – AC were calculated to be 16.11 nm and 36.13 nm respectively that were obtained from XRD pattern. The adsorption capacity of Ag NPs – AC was analysed and the optimum conditions were determined using different parameters which are the Ag NPs - AC ratio (1.0 g), contact time (240 min), adsorbent dosage (30 mg) and pH of CV dye (10). The highest percentage removal of CV dye using Ag NPs – AC was recorded up to 97% at 240 min with 30 mg dosage. Ag NPs – AC as adsorbent is a promising advanced material in removing water pollutants with viable conditions and can applied in the wastewater treatment industry.

*Keywords: Silver nanoparticles, activated carbon*
*green synthesis, dye adsorption*

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## 1. Introduction

Water pollution associated with dye pollutants are becoming worsen since the annual worldwide production of commercial dyes are expected more than 100,000 types [1]. Even worse, it is also reported that the worldwide industrial effluent estimated 280,000 tons of textile dyes are being discharge every year [2]. The existence of dyes and paints from various industries such as textiles, plastics, cosmetics, food and petroleum refineries contain heavy loads of mixed colours from its discharged effluent that reflect toxicity to aquatic life [3]. Moreover, the presence of dyes in wastewater provide significant source of water pollution because even at low concentration the dye waste is visible and can harm aquatic life [4]. For instance, high concentration of crystal violet (CV) dye in water can affect the oxygenation and photosynthesis capacity in the water [5].

Generally, there are three methods in removing dye which are biological, chemical and physical methods. Biological methods promote microorganism such as algae, yeast, fungi and bacteria that can transform dye into less harmful forms [6]. Meanwhile, chemical methods includes photocatalytic degradation, ozonation and electrochemical treatment while physical method include precipitation-flocculation, adsorption, ion exchange and membrane filtration [4]. Among all the methods, adsorption process in removing dye pollutants shows promising results because it is low cost, ease in operation, can eliminate all type of contaminants, flexible, simple and insensitivity towards toxic pollutants [7].

Recently, various nanoparticles that had been studied for the application of dye removal in water and wastewater remediation. The application of adsorption process commonly used nanoparticles such as carbon-based, metal, ceramic, polymeric and lipid-based nanoparticles [8]. Among the nanoparticles involved, silver nanoparticles (Ag NPs) had successfully remove dye for water and wastewater treatment due to its nano-size that provide high surface area to volume ratio in adsorbing dye [9]. However, the drawback of using Ag NPs alone in dye treatment is aggregation of silver (Ag) ions in water that may cause hazard towards environment. Hence, Ag NPs incorporated with surface adsorbent can reduce the limitations and provide even higher adsorption capacity due to great potential of both Ag NPs and adsorbent such as activated carbon (AC) which is low cost and high porosity properties and widely used adsorbent to remove pollutants [10].

Silver nanoparticles loaded activated carbon (Ag NPs – AC) had shown great performance in removing dye pollutants. For instance, CV dye was removed with adsorption capacity of 82.2 mg/g and the reusability of this materials in optimum condition was 7 consecutive times [11]. Moreover, it is reported that Ag NPs – AC capable of removing Sudan red 7B with a maximum adsorption capacity of 90.909 $mg.g^{-1}$ and low removal time [12]. Hence, the use of Ag NPs – AC is very promising with potential benefits in removing dye by adsorption process for water and wastewater treatment application and promoting higher active sites of AC for better adsorption process.

Green synthesis method in producing Ag NPs via plant extract are eco-friendly, rapid, cost-effective and non-pathogenic which drawn attention of researcher in replacing sodium borohydride, hydrazine and sodium citrate as reducing and stabilizing agents. This provides minimal waste, reduced energy use, renewable materials and low risk methods [13]. Butterfly pea or *Clitorea Ternatea* (CT) flower belongs to the family of Fabaceae that are available in tropical Asia such as India, Thailand, Myanmar, Indonesia, Philippines and Malaysia [14]. In this research, CT flower extract was used as the stabilizing agent in producing Ag NPs. It contains high amount of anthocyanin pigments compare to other flowers which is the main components in the green synthesis process [15].

## 2. Materials and Methods

All chemicals and materials used were analytical grade with silver nitrate ($AgNO_3$), sodium hydroxide (NaOH), hydrochloric acid, (HCl) and CV dye powder were purchased from R&M Chemicals, Malaysia. Meanwhile, AC powder was purchased from Chemiz, Malaysia with no further heating or activation. Fresh CT flowers were collected at a community farm near Keramat, Kuala Lumpur, Malaysia. The flowers were grown under no specific care of watering and fertilizer since the flowers grew freely and abundant without maximal care. All aqueous solutions were used with deionized water.

### *2.1. Synthesis and characterization of Ag NPs – AC*

The collected CT flowers were double wash with tap water and deionized water to remove dirt and sand at the surface of the flowers. Then, the flowers were dried for 2 hr at 70°C in an oven. The dried CT flowers were crush into powder using conventional home blender (Phillip Blender Core Series 5000) and stored in an airtight container to ensure no moisture and preventing mold and fungus grown. Next, 0.05 g of powdered CT flowers were mixed with 100 ml of deionized water with continuous stirred at 500 rpm for 1 hr in room temperature to produce CT flower extract.

Then, the aqueous extract was centrifuged (KUBOTO Tabletop Micro Centrifuge Model 3300) for 30 min at 4000 rpm and filtered using Whatman grade 1 filter paper. The flower extract was then transferred into a centrifuge tube for further use and stored in a freezer. 1 ml of 0.1 M NaOH were used to alter 20 ml of prepared CT flower extract to pH 10. Next, the extract was stirred for 10 min at 500 rpm and mixed with 4 ml of 3 mM $AgNO_3$ at 50°C for 30 min to produce Ag NPs solution.

Nanocomposite of Ag NPs – AC were prepared by added 4 ml of 3 mM $AgNO_3$ with mass variation of AC (0.25, 0.5, 0.75 and 1.25 g) and stirred for 10 min at 500 rpm. Then, CT flower extract of pH 10 were mixed with the solution at 50°C for 30 min. Then, the solution was centrifuged, sonicated and washed for 3 consecutive times. Finally, the produce Ag NPs – AC were dried at 60°C for 24 hr and ready to be used as adsorbent powder.

Ag NPs was characterized using UV – Visible Spectroscopy (UV – Vis) (Shimadzu UV – 2600) to study the formation of Ag NPs via green synthesis method. Furthermore, Ag NPs and Ag NPs – AC were characterized with X – ray diffraction (XRD) using XRD-Empyrean (PAN analytical, United Kingdom). XRD analysis pattern at 2θ angle from 20° to 80° were analysed for both materials.

### *2.2. Batch adsorption of CV dye*

The batch adsorption of CV dye started with the preparation of 200 ppm or 200 mg/L CV dye solution. 50 ml of CV dye solution was transferred into a 100 ml conical flask for further used for every parameter. Next, 30 mg of Ag NPs – AC was used initially and mixed in the conical flasks that were place on an orbital shaker (IKA Shakers KS 260 basic) at a constant 300 rpm with different parameter such as Ag NPs - AC ratio (0.25, 0.5, 0.75 and 1.25 g), contact time, adsorbent dosage (10, 20, 30 and 40 mg) and CV dye pH (3, 7, 9 and 10). Analysis of initial and final concentration of CV dye were analysed using UV-Vis and calculated using Eq. 1 below to know the percentage of removal by using Ag NPs – AC as adsorbent where Abs = Absorbance.

$$\%\ \text{CV Dye Removal} = \frac{\text{Initial Abs} - \text{Final Abs}}{\text{Initial Abs}} \times 100\% \qquad (1)$$

## 3. Results and Discussion

### *3.1. Synthesis of Ag NPs – AC*

The green synthesis of Ag NPs was successfully conducted using CT flower extract. The CT flower extract acts as stabilizing agent for $AgNO_3$. Anthocyanin pigments that present in the CT flower is the key element in the green synthesis of Ag NPs that replaced conventional reducing agent.

The synthesis route is simple and fast as shown in Figure 1 where the total time from preparing the flower extract to producing Ag NPs was only 90 min. Besides, the heating used was low which was 50°C to fasten the reaction. In this study, 0.1 M of NaOH was used to alter the flower extract to alkaline medium. It is because it took longer time to produce Ag NPs in neutral medium. This finding was also supported by Khwannimit *et al.* [16] that uses NaOH to fasten the reaction. Observation directly by naked eye clearly shows the colour changes from light blue to dark brown indicating the formation of Ag NPs and reduction of $Ag^+$ to $Ag^0$ as shown in Figure 2. Furthermore, Ag NPs – AC was successfully synthesized from Ag NPs solution and AC with mass variation ranging from 0.25, 0.5,

0.75 and 1.25 g via in-situ method. Ag NPs – AC was produced in the form of black powdered mainly came from the AC. Naked eye observation shows that the Ag NPs – AC contain very fine shiny silver particles which is the Ag particles. This confirm the loading was successful and the technique was valid.

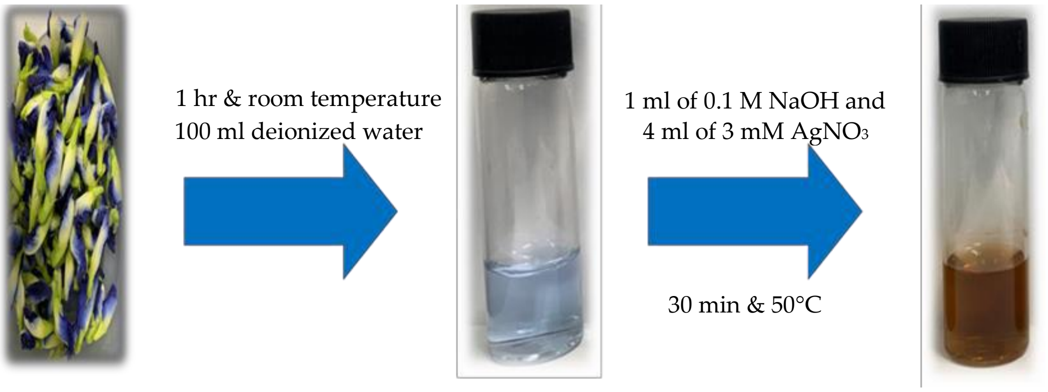

**Figure 1.** Green synthesis of Ag NPs from CT flower extract.

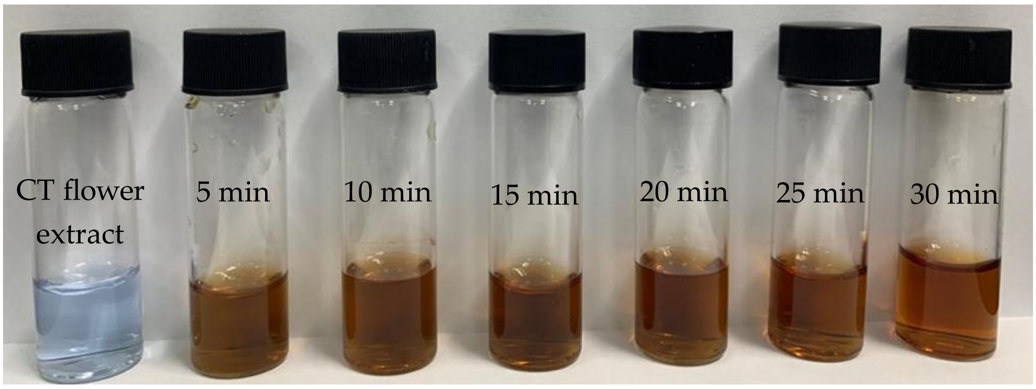

**Figure 2.** Complete formation of Ag NPs in 30 min.

### *3.2. UV-Visible spectroscopy analysis*

UV-Vis analysis was carried out to study the formation of Ag NPs via the green synthesis method. Figure 3 shows the UV-Vis graph for Ag NPs solution with peaks at 408 nm. The UV-Vis graph pattern from this study correspond with the research done by Khwannimit *et al.* [16] which confirm the formation of Ag NPs but with different peak value which is 430 nm that indicates even smaller nanoparticles produces. This is because the optimum condition for the green synthesis of Ag NPs which range from the temperature, pH, reaction time, $AgNO_3$ concentration and volume ratio of $AgNO_3$ to the CT flower extract were not analysed in this study. Analysing these parameters can obtain the optimum synthesis condition in producing smaller Ag NPs. This is because loading smaller Ag NPs into AC need to be achieved since it can increase the adsorption capacity and reactive center of AC compared to pure AC [11].

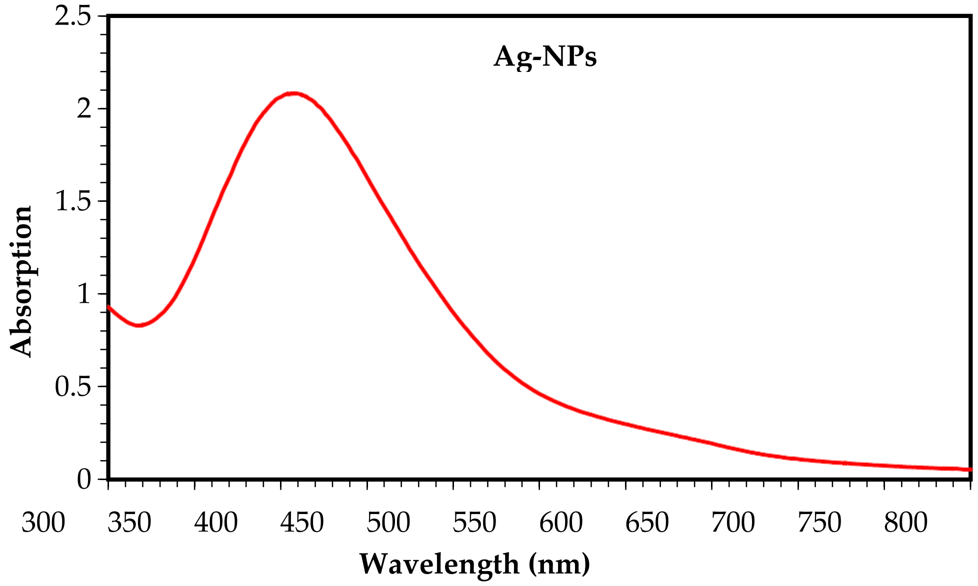

**Figure 3.** UV-Vis graph for the formation of Ag NPs.

### *3.2. XRD analysis*

The nanostructured and crystal of Ag NPs and Ag NPs – AC was analysed by using XRD. The XRD graph as shown in Figure 4 shows two graph for Ag NPs and Ag NPs – AC respectively. Four peaks values for Ag NPs – AC at 2θ of 38.13°, 44.28°, 64.32° and 77.46° were identified. Meanwhile four peak values for Ag at 2θ of 38.19°, 44.43°, 64.57° and 77.43° corresponding with the face-centred cubic structure (FCC) of Ag [17] and its crystalline nature with sets of lattice planes of (111), (200) and

(311) [18-21]. Furthermore, the crystallite size of Ag NPs and Ag NPs – AC was calculated based on the XRD pattern using Scherrer's equation as shown in Eq.2 where D = average crystallite size, k = constant, λ = wavelength, β = full width at half maximum (FWHM) and θ = Bragg angle [22-24]. According to the equation, k = 0.94 and λ = 1.54060. The average of crystallite size of Ag NPs and Ag NPs – AC for this work is 16.11 nm and 36.13 nm respectively.

$$D = \frac{k\lambda}{\beta\cos\theta} \qquad (2)$$

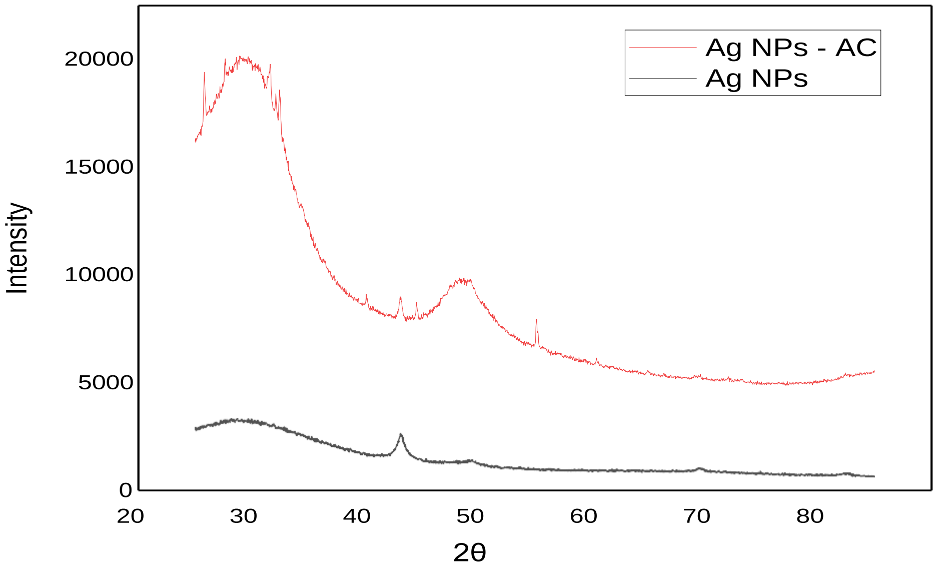

**Figure 4.** XRD analysis for Ag NPs and Ag NPs – AC.

### *3.3. Batch adsorption*

### *3.3.1 Effect of Ag NPs - AC ratio*

The first parameter studied in this research is the effect of AC mass variation during the synthesis of Ag NPs – AC. The mass variation was ranging from 0.25, 0.5, 0.75 and 1.25 g with Ag NPs that was kept constant since the Ag NPs was in aqueous solution form. The experiment conducted is to study the effect of Ag NPs – AC ration as compared to pure AC. Figure 5 shows the effect of pure AC and variation of Ag NPs – AC ration on the percentage of CV dye removal. From the graph, pure AC removed around 70.9% of CV dye.

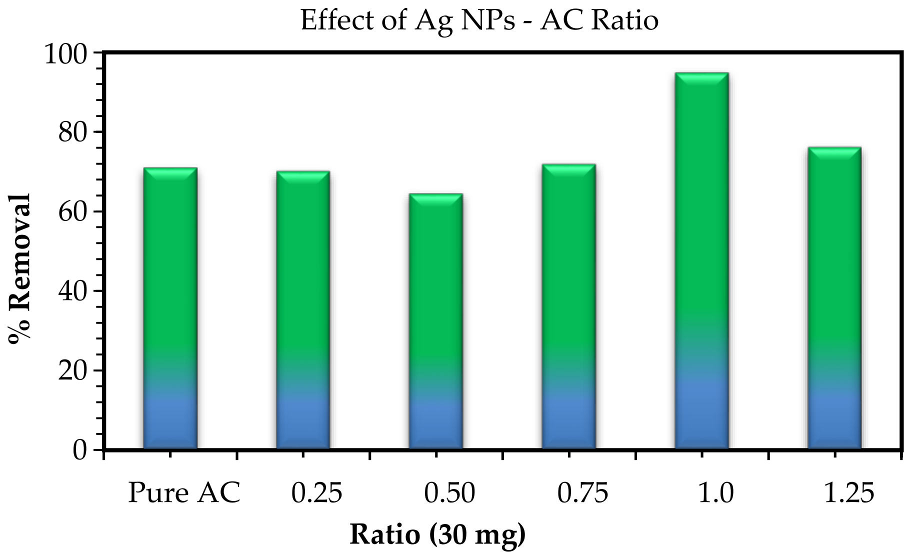

**Figure 5.** Effect of Ag NPs - AC ratio

However, the data also shows that for AC ratio of 0.5 and 0.25 g the percentage removal was lower than pure AC indicating that the ratio is not suitable for Ag NPs – AC formation mainly because the amount of AC is insignificant or less since AC is the main adsorbent in removing CV dye. Meanwhile, for ratio from 0.75 until 1.25 g shows promising results as it is higher than pure AC. In this experiment, 1.0 g ratio shows excellent results as it can remove up to 94.9% of CV dye that specify the ratio is acceptable for the synthesis of Ag NPs - AC. Hence, 1.0 g ratio is the most suitable for the removal of CV dye.

### *3.3.1 Effect of adsorption contact time*

The effect of contact time is very important to design an economical wastewater treatment. This is because longer time in removing the pollutants will cause more utilities and energy need to be used. Therefore, having a shorter contact time for the process to be carried out can give high impact and significant changes in a wastewater treatment for dye removal. The data and information shows that the adsorption was rapidly increased from 10 to 90 min indicating more active sites available in AC to remove CV dye as shown in Figure 6. Meanwhile, from 120 to 240 min the removal percentage were slow which indicates that the active sites were decreasing upon increasing of time. It is also because of concentration gradient and diffusion rate that are lowering in increasing of contact time [25]. At 240 min, the percentage removal was up to 97.7% which correspond to nearly all CV dye was adsorb with the observation of colorless solution. Therefore, the optimum contact time in removing CV dye is 240 min where the adsorption above the time will further decrease because of the unavailability and limited active sites present in the adsorbent [26].

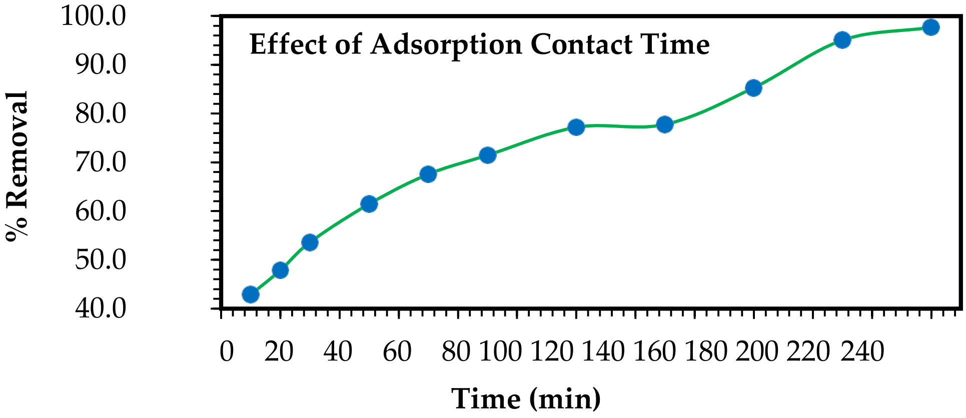

**Figure 6.** Effect of adsorption contact time.

### *3.3.1 Effect of adsorbent dosage*

The effect of adsorbent dosage is crucial since it is relatable to the economic and environmental evaluation. This is because the more the dosage, the more the cost and effect in environment. This parameter tested the adsorbent dosage of Ag NPs – AC ranging from 10 – 40 mg. The preliminary hypothesis indicates that the more the adsorbent dosage, the higher the removal percentage. Hence, Figure 7 approves the hypothesis since 30 and 40 mg dosage shows high adsorption capacity. In this experiment, the adsorbent dosage that shows promising results were 30 and 40 mg with removal percentage of 97.7% and 98.1% at 240 min respectively. Since, the optimum contact time is 240 min, the adsorbent dosage was chosen based on 240 min contact time from previous parameter [26]. However, adsorbent dosage of 40 mg was not choose as the best condition of CV removal since the adsorption capacity was not significantly high compare to 30 mg. Therefore, 30 mg was choosing as the best condition in removing CV dye since the adsorption capacity is high and to maintain less adsorbent dosage, low cost and effect towards the environment.

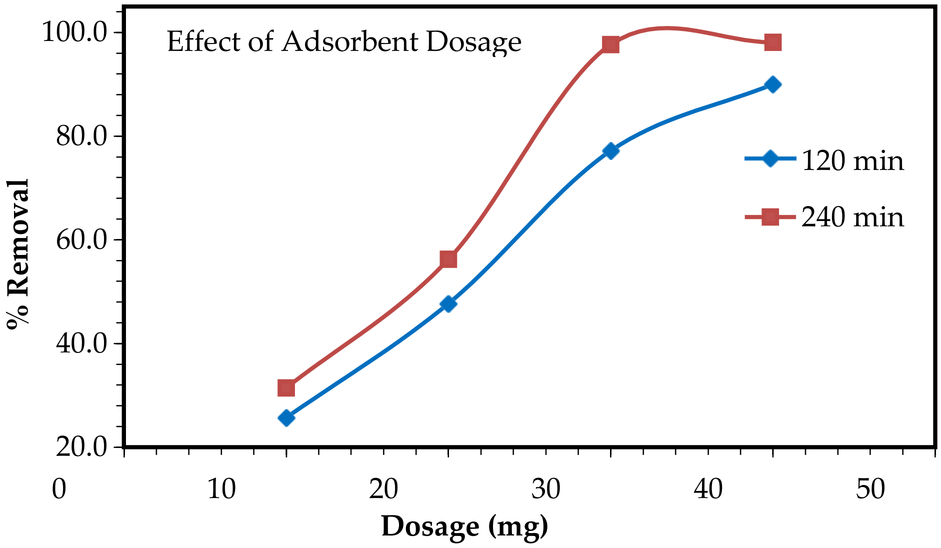

**Figure 7.** Effect of adsorbent dosage.

### 3.3.1 *Effect pH of CV dye*

The last parameter is the effect of dye pH which is to know the best condition of the solution for better adsorption capacity study. In this study, pH of CV dye ranging from 3, 7, 9 and 10 were tested in order to study the best medium in which Ag NPs – AC can absorb CV dye with the highest capacity. The CV dye pH was adjusted by using NaOH and HCl for alkaline and acidic medium respectively with initial pH was 5. The data shows that the best condition is the alkaline medium since pH 9 and 10 shows the highest adsorption capacity for Ag NPs – AC as shown in Figure 8 with similar result reported by AbdEl-Salam, Ewais [11]. Meanwhile, in acidic medium which is at pH 3, the adsorption was only 84% compared to alkaline medium that can reach to 100% removal. This is because in alkaline medium, strong electrostatic attraction between cationic CV dye and negative charge of Ag NPs – AC that results with increase in CV dye adsorption [25]. Hence, pH 9 and 10 is the best medium in removing CV dye but pH 10 was chose in this study because the adsorption process was rapid for the first 5 min compared to pH 9.

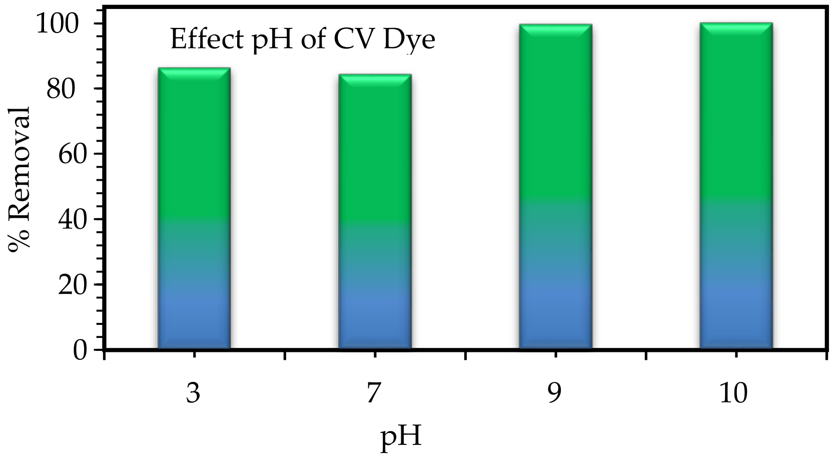

**Figure 8.** Effect pH of CV dye.

## 4. Conclusions

Water pollution causes by dye from dyeing industry need to be tackle with cost-effective and sustainable methods. In this present study, adsorption process was introduced in removing CV dye by using Ag NPs – AC that promotes high surface area and porous structure. Ag NPs – AC as adsorbent was produced by plant mediated synthesis of Ag NPs using CT flowers that promotes stabilizing agents and loaded with AC. The Ag NPs was characterized using UV – Vis which correspond at 408 nm. Moreover, The XRD analysis for Ag NPs and Ag NPs – AC provide significant information regarding crystallite size which is 16.11 nm and 36.13 nm. The adsorption capacity of Ag NPs – AC were determined by analysing the effect of Ag NPs - AC ratio (1.0 g), adsorption contact time (240 min), adsorbent dosage (30 mg) and pH of CV dye (10). In future, the study regarding removal of dye using Ag NPs - AC can be improving by implementing reusability or regenerating study of the adsorbent to fully maximise the adsorption capacity. Furthermore, the study regarding the adsorbent waste management need to be analysed to ensure the water will not be polluted with the adsorbent. Finally, the study on the aggregation of Ag ions in aqueous solution can be determine to analysed the health and hazard potential towards human and aquatic life.

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