

# Binary Adsorption of Textile Dyes onto Zwitterionic Adsorbent Coating: Performance Study

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**Abstract** Textile wastewater is considered as the most polluted of all industrial sectors, both from the amount produced and the effluent composition. Most of the previous researches concentrates on extracting dyes from water in a single solution. In real applications, the colored effluents contain more than one removable component. In this study, the adsorption of two textile dyes (Acid Red 1 and Brilliant Green) was investigated in a binary mixture. An adsorbent coating with zwitterionic interfaces was introduced as a new approach for wastewater treatment, named as zwitterionic adsorbent coating (ZwitAd). The derivative spectrophotometry for each dye was used to determine the precise wavelength. Consideration was given to the effect of pH, contact time, initial dye concentrations, and temperature. The findings show that the ZwitAd has a strong potential to simultaneously remove both dyes. Equilibrium was achieved within 300 min and to obtain a higher percentage removal of Acid Red Brilliant Green, ARBG, the optimal condition has been achieved. The higher percentage removal for AR1 and BG was reported at 92.14% and 90.18% respectively at 10BG-40AR (ppm) initial concentration.

**Keywords:** Binary Adsorption, Zwitterionic Adsorbent Coating, Cationic Dye, Anionic Dye

## 1. Introduction

The wastewater from textile plants is rated as the most polluted of all industrial sectors, taking account both the amount generated and the composition of the effluent involved [1]. Furthermore, the increased demand of textile products and the proportional increase in their production be one of the major sources of severe pollution problems in current times. In addition with the trending use of synthetic coloring nowadays, the contribution of hazardous wastewater also increased [2]. A lot of water is involved in the dyeing process and not all places have efficient ways to disinfect the water until it goes back into the environment [3].

A dye is a compound that has multiple colours and usually used in solution as it is easy to be fixed with fabric and other material. Dyes are categorized by application and chemical structure and made up of a group of atoms known as chromophores, responsible for the colour of the dye. These centres containing chromophores are based on various functional groups, such as azo, anthraquinone, methine, nitro, arilmethane, carbonyl, and others. Furthermore, electrons that remove or donate substituents to produce or amplify the colour of chromophores are called auxochromes. Amine, carboxyl, sulfonate, and hydroxyl are the most common auxochromes [3-5]. The dyes can maintain to the compatible surface with a solution by forming covalent bonds or complexes with salts or metals through physical adsorption or mechanical retention. More than 100,000 synthetic colors — cationic, anionic, and dispersive — have been commonly used in the afore mentioned industries generating volumes of

wastewater annually [3]. These are not easy to biodegrade since it developed highly stable molecules, which are made to withstand degradation by chemical, biological and light penetration, as well as their multifarious chemical structure.

Before the wastewater are allowed to discharge into the water stream, there are several methods applied to remove the highly toxic organic compound from polluted water, such as coagulation, precipitation, adsorption, ion exchange and reverse osmosis [5-7]. From those method, the adsorption process is important and should be carried out optimally to serve its role as a part of the water treatment process. The adsorption process is a surface phenomenon where a solution containing absorbable solutes contact with a highly porous solid structure, liquid-liquid intermolecular forces of attraction cause some of the solute molecules from the solution to be concentrated or deposited at the solid surface [5,8].

Previous work on dye adsorption was performed tremendously in single dye systems, and records of scanty analysis were available in binary dye systems. Multiple components of dyes typically coexist in actual industrial wastewater systems. This multiplicity in real applications can have an antagonistic or synergistic effect on the dye adsorption process. Therefore, it is imperative to investigate adsorption of dye both in single and multiple systems especially by evaluating the efficient adsorbents to work with [9-11].

In real applications, the colored effluents contain more than one removable component. Previous studied was investigated a binary mixture involved two textile dyes Red Bemacid-ETL (RB-ETL) and Bezaktiv Türkei Blue-VG (BTB-VG). The derivative spectrophotometry for each dye was used to determine the precise wavelength. Consideration was given to the effect of pH, contact time, and initial dye concentrations. The results show that diatomite has good potential to simultaneously eliminate both dyes [12]. Another research was simultaneous analyzed Basic Blue 41 and Basic Yellow 28 in binary system using Persea Americana Nut Carbon (C-PAN) and Ziziphus Mauritiana Nuts Carbon (C-ZMN) as two forms of activated carbon adsorbents. A competitive adsorption was observed between the two cationic colours and it proved that adsorption was an efficient method for decoloring simple textile dyes [13].

Therefore, this study has investigated a newly type of adsorbent coating named as ZwitAd. Previous studied shown its potential in eliminating 2 types of dyes (cationic and anionic) in single adsorption process [14,15]. In order to prove the efficiency of ZwitAd in removing both dyes by binary adsorption, further investigation was carried out to evaluate the performance in terms of initial concentration, solution pH and temperature of binary dyes.

## 2. Materials and Methods

### 2.1. Materials

Poly-epichlorohydrin-dimethylamine (EPIDMA) (50 wt. % in H<sub>2</sub>O) and acrylic polymer emulsion (APE) were purchased from NHA Scientific Resource, Cyberjaya, Malaysia. The EPIDMA is a type of cationic polyelectrolyte and has been used as a surfactant to synthesize adsorbent coating. Bentonite, a mineral clay was acquired from Modern Lab Sdn. Bhd., Malaysia. Sodium hydroxide, NaOH and hydrochloric acid, HCl were both obtained from Merck, Malaysia. Cotton cloth was purchased from Kedai Kain 1 Malaysia, Parit Buntar, Malaysia. Deionized water was used to prepare the adsorbent coating solutions. Brilliant green (BG), C<sub>27</sub>H<sub>34</sub>N<sub>2</sub>O<sub>4</sub>S ( $\lambda_{\max}$  = 531 nm) and Acid Red 1 (AR1), C<sub>18</sub>H<sub>13</sub>N<sub>3</sub>Na<sub>2</sub>O<sub>8</sub>S<sub>2</sub> ( $\lambda_{\max}$  = 624 nm) dyes were supplied from Chemolab Supplies Sdn. Bhd and used as an adsorbate.

### 2.2. Preparation of zwitterionic adsorbent coating strip

The complete preparation method of ZwitAd was briefly explained in previous studied however with few modification [14,15]. In general, 1 g of bentonite powder was added slowly to 20 mL of deionized water in a Schott bottle. The bottle is then closed tightly, and the bentonite is left to swell up. After 3 hours, a magnetic bar is added into the bottle and the solution is left to mix on a magnetic stirrer at 300 rpm for another 6 hours. This step is crucial to ensure the complete swelling of bentonite. Next is to add 2 wt. % EPIDMA solution into the swollen bentonite. Before that, the EPIDMA solution is first prepared by dropping 2 mL of EPIDMA into 48 mL of deionized water. The solution is then left to stir on a magnetic stirrer at 300 rpm for 6 hours.

To produce the final ZwitAd solution, acrylic polymer emulsion (APE), swollen bentonite and EPIDMA solution were mixed together with a mass ratio of 1:2:4 respectively. In a new Schott bottle, 25 mL of bentonite suspension, 50 mL of 2 wt.% EPIDMA solution and 12.5 g of APE is added. This solution is then left to stir on the magnetic stirrer at 300 rpm for another 8 hours to allow the milky greyish-white solution formed and completely dissolve. This is the final adsorbent coating suspension and further signed as ZwitAd solution.

For the preparation of ZwitAd strip, cotton cloth is first cut into strips of 20 cm in length and 5 cm in width. It is then washed to remove dirt or particles that might affect the result of the experiment. After drying the strips of cloth in the oven, 3 g of the adsorbent coating suspension is manually applied onto both sides of each cloth strip by manually painting using a paint brush. The cotton strips were then hung in the oven with the help of cloth pegs for another 3 hours to evaporate excess water from the suspension. As the cotton strips get dried, continuous evaporation of water causes the particles of the adsorbent

coating get closer to each other and leads to deformation of the particles. The individual particles disappear while the polymer chains diffuse across the particle borders and become entangled with one another, eventually forming an even coating across on the cotton strip [16]. The ZwitAd strip was rinsed using tap water to remove residue/uncoated coating. The sample was dried again in the oven overnight and the dried product ready for further treatment.

### 2.3. Preparation of Stock Solution

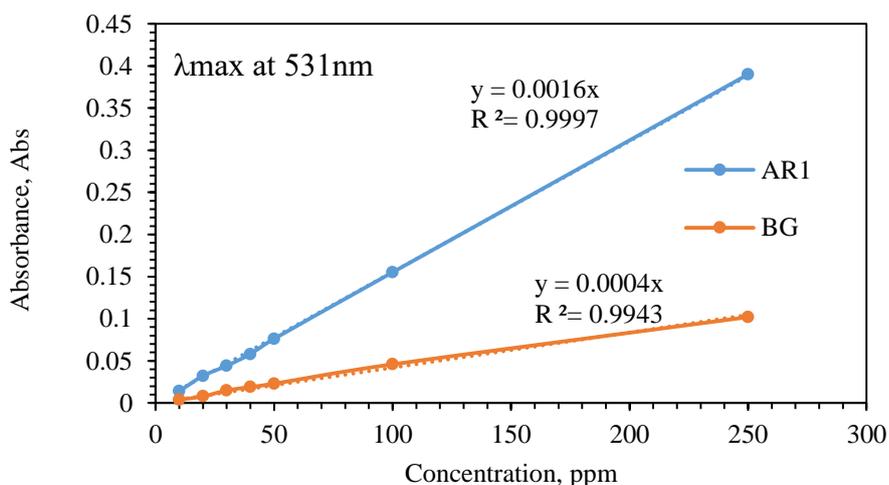
The stock solution of Acid Red 1 (AR1) and Brilliant Green (BG) was first setup. The 1000 ppm of stock solution of both dyes were prepared by dissolving 1 g of AR1 and BG dye powder into 1000 mL distillate water. These two dyes were prepared separately in the volumetric flask and were stirred using magnetic stirrer until the dyes were dissolved completely without left any agglomeration. Then, the stock solution for these two dyes was diluted based on the required initial concentration between 10 mg/l to 100 mg/l for adsorption studies. Further binary concentration of dyes mixture were diluted according to these concentration (50-50 ppm AR-BG, 100-100 ppm AR-BG, 10-40 ppm AR-BG, 10-90 ppm AR-BG, 40-10 ppm AR-BG and 90-10 ppm AR-BG).

### 2.4. Determination of Maximum Wavelength of Binary Dyes

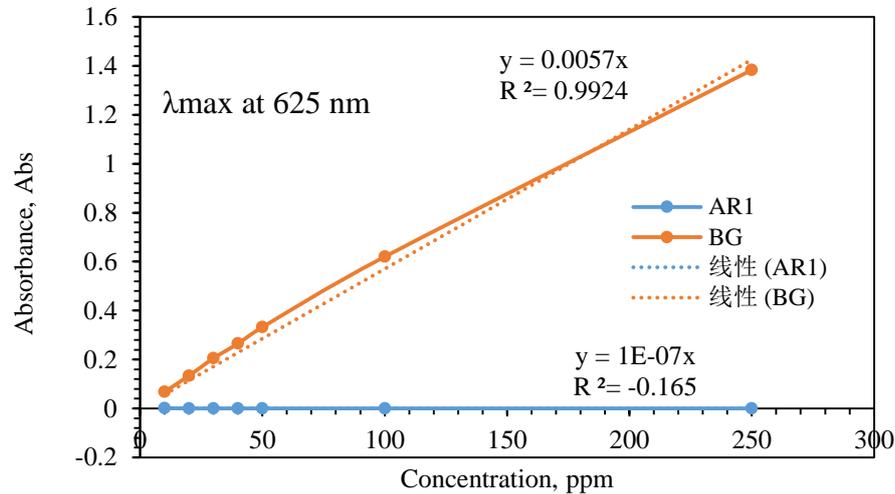
The different binary initial concentration of AR1 and BG dyes was prepared according to the concentration of 30AR-30BG ppm, 40AR-40BG ppm and 50AR-50BG ppm. This procedure is essential to determine either the solution will react with each other since both dyes containing different charge. As been checked based on single dyes, the maximum wavelength of Acid Red 1 was obtained at 531 nm, while Brilliant Green was 625 nm. The wavelength of dyes was analyzed using UV-Vis spectrophotometer (UV-1800, Shimadzu Corporation, Kyoto, Japan). The range of wavelength was set at 300 to 800nm.

### 2.5. Calibration Curve

The standard calibration curve of AR1 and BG were prepared by diluting 1000 ppm dyes into 10 mg/l, 20 mg/l, 30 mg/l, 40 mg/l, 50 mg/l, 100 mg/l and 250 mg/l initial concentration. To evaluate the absorbance value at full wavelength, the samples value were taken at both wavelength of 531 nm and 625 nm. The standard calibration curve measurement was repeated until the obtained regression coefficient,  $R^2$  was nearest to 1. Therefore, to measure the concentrations of each dye in binary solution, four calibration curves were constructed and illustrated in [Figure 1](#) and [Figure 2](#). [Table 1](#) shown the calibration constant value of AR1 and BG dyes.



**Figure 1.** Calibration curve of AR1-BG at 531nm



**Figure 2.** Calibration curve of AR1-BG at 625nm

**Table 1.** The calibration constant value

Measured at $\lambda_{\max} = 531 \text{ nm}$		Measured at $\lambda_{\max} = 625 \text{ nm}$	
kR1	0.0016	kR2	0.0000
kB1	0.0004	kB2	0.0057

## 2.6. Single Adsorption Isotherm Experiment

At first, the isotherm adsorption was studied for a single component solution. The volume of AR1 and BG were set at 200 mL in each beaker, with initial dye concentrations of 10 mg/L, 20 mg/L, 30 mg/L, 40 mg/L, 50 mg/L, 100 mg/L and 250 mg/L. The amount of adsorbent coating was fixed for all the experiments. The 100 cm<sup>2</sup> cotton cloth was placed and clipped at the upper inside of the glass beaker and the strip was immersed in a dye solution. The magnetic bar was placed at the bottom of the beaker and the dye solution was stirred gently using the stirrer at 350 rpm, for 5 hours at room temperature. The changes in dye concentration in each solution was determined by using a spectrophotometer to evaluate the absorbance value. The concentration of each dye was measured based on the maximum wavelength of each dyes. The adsorption capacity at equilibrium condition,  $Q_e$  (mg/g) was calculated using Equation 1 and the dye removal percentage using Equation 2 [17].

$$q_e = (C_o - C_e) \frac{V}{W} \quad (1)$$

$$\% \text{ Removal} = \left( \frac{C_o - C_e}{C_o} \right) * 100 \quad (2)$$

Where,  $C_o$  = Initial dye concentration (mg/L),  $C_e$  = Final dye concentration (mg/L),  $W$  = Weight of adsorbent coating,  $V$  = Volume of dye solution (L)

## 2.7. Binary Adsorption Isotherm Experiment

In binary system, the dye removal experiments were performed by mixing both AR1 and BG dyes in 250 mL beaker. The concentration of each dye was depending on the experiments. The experiment was carried out using five different pH (3, 5, 7, 9 and 11), while the temperature was varying at 30 °C, 45 °C, 60 °C and 75 °C. Wider range of pH has been chosen as dyes have a variety of substances ranging from acidic and alkaline material. Therefore, to determine the optimum initial pH for this binary solution, the pH was set from 3 to 11. To investigate the effect of initial dye concentration, these two dyes were mixed at different initial concentrations (50-50 ppm AR-BG, 100-100 ppm AR-BG, 10-40 ppm AR-BG, 10-90 ppm AR-BG, 40-10 ppm AR-BG and 90-10 ppm AR-BG). The adsorption processed was carried out similar as single adsorption procedure.

### 2.8. Measurement of Dye Concentration in Binary Solution

The common procedure to identify the dye concentration in their mixture is using UV-Vis spectrophotometer. To this end, a linear relationship was applied between absorbance (A) and dye (C) concentration (mg/L) given by Beer-Lambert law in Equation 3 [18].

$$A = KC + E \tag{3}$$

Where, A = Absorbance of light at maximum wavelength ( $\lambda_{max}$ ), K = Absorbance coefficient (slope of linear relation), C = Concentration of dye solution (mg/L), and E = Intercept of a linear relation.

For binary systems, the total absorbance A1 at  $\lambda_{1max}$  is the sum of the absorption of the individual AR1 and BG components and similarly for A2 at  $\lambda_{2max}$ , which can be written as Equation 4 and Equation 5 respectively.

$$A1 = kR1 * CR + kB1 * CB \tag{4}$$

$$A2 = kR2 * CR + kB2 * CB \tag{5}$$

The above equations result in Equation 6 and Equation 7 respectively. Equations 6 and 7 state the concentration values for each variable, CA (AR1) and CB (BG).

$$CA = \frac{(kR2 * A1 - kB1 * A2)}{(kR1 * kB2 - kR2 * kB1)} \tag{6}$$

$$CB = \frac{(kR1 * A2 - kR2 * A1)}{(kR1 * kB2 - kR2 * kB1)} \tag{7}$$

Where, kR1 = Calibration Constant for AR1 at wavelength 531 nm, kB1 = Calibration Constant for BG at wavelength 531 nm, kR2 = Calibration Constant for AR1 at wavelength 625 nm, kB2 = Calibration Constant for BG at wavelength 625 nm

## 3. Result and Discussion

### 3.1. Maximum Wavelength of Binary Dyes

The maximum wavelength of AR1 and BG mixed dyes can be identified in the ARBG plotted in Figure 3. From the graph, the maximum wavelength for binary dyes have remained at 531 nm and 625 nm peaks for AR1 and BG, respectively. The gradient of concentrations (30, 40 and 50 ARBG) were analyzed to confirm that there will no deviation of peak at lower or higher combined initial concentration throughout the adsorption processed. Figure 4 displayed mixtures of dyes between BG and AR 1 dyes at concentrated solution.

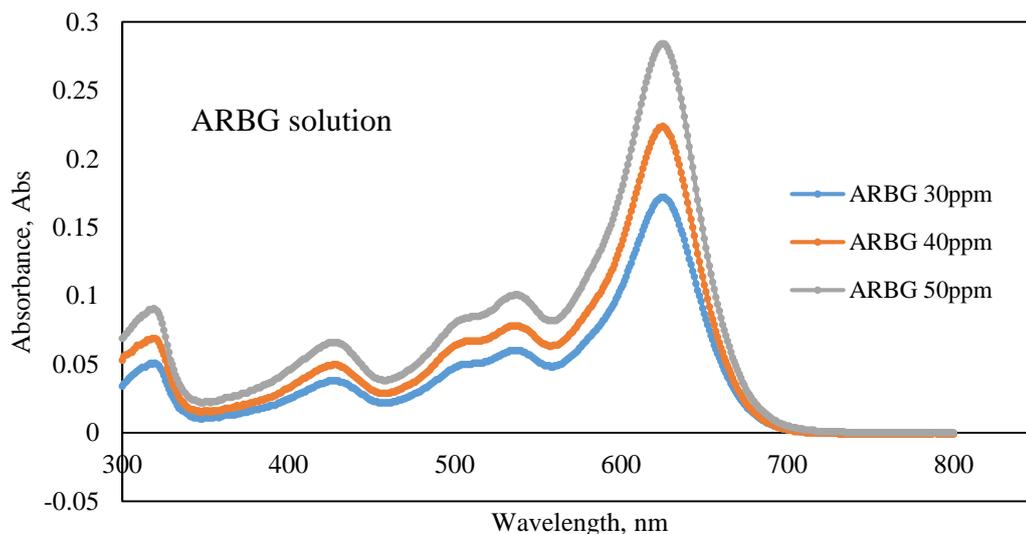
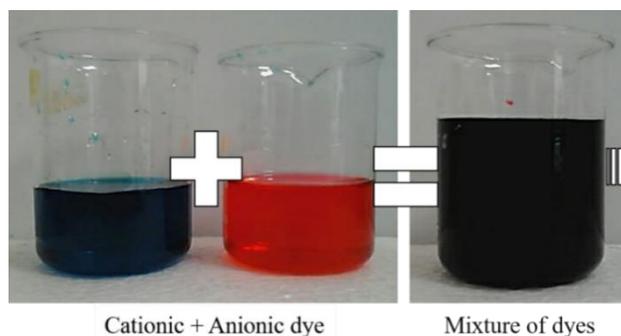


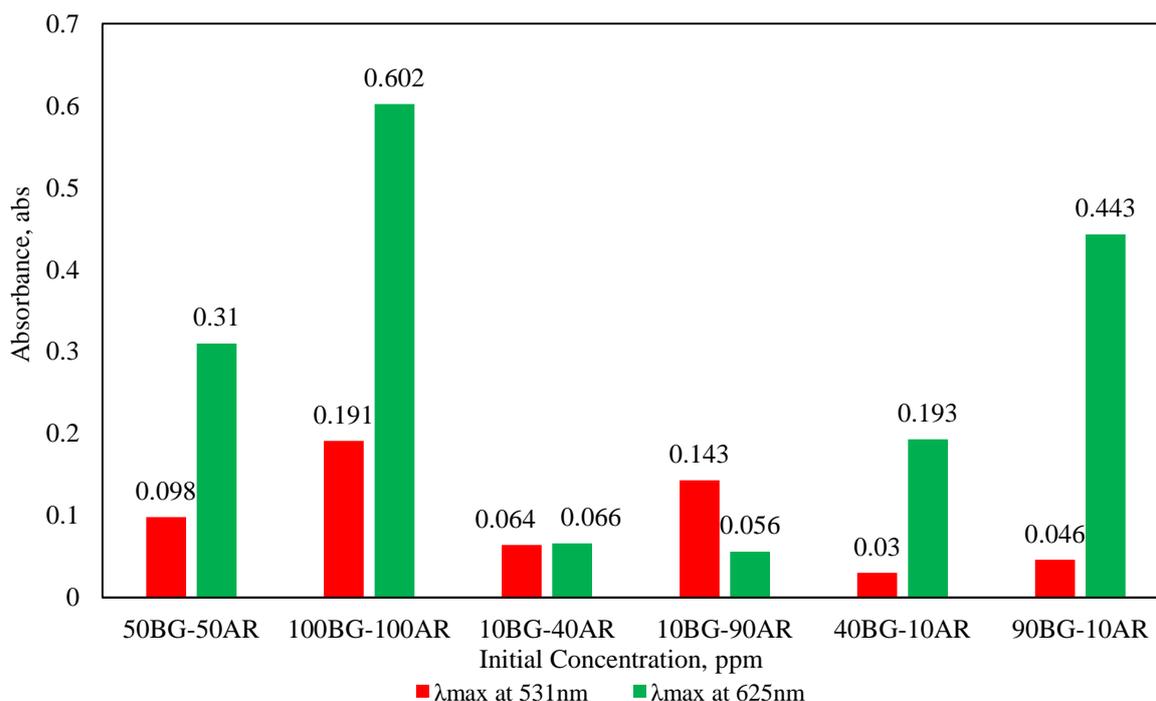
Figure 3. The maximum wavelength for ARBG solution



**Figure 4.** Digital photograph of BG (cationic) and AR1 (anionic) as a single and mixtures of dyes

### 3.2. Effect of Initial Concentration

The initial concentration of dyes is an important factor to be studied in adsorption process because most polluted wastewaters typically contain specific concentrations of dyes [19]. Figure 5 shown the absorbance values at different initial concentrations of ARBG solution for two maximum wavelengths at 531 nm and 625 nm. It was shown that at 100BG-100AR (ppm), the highest absorbance value was 0.191 abs for  $\lambda_{max}$  at 531 nm and 0.602 nm at  $\lambda_{max}$  of 625 nm. The lowest absorbance value was recorded at 10ppm of AR1 (0.03 abs) and BG (0.56).

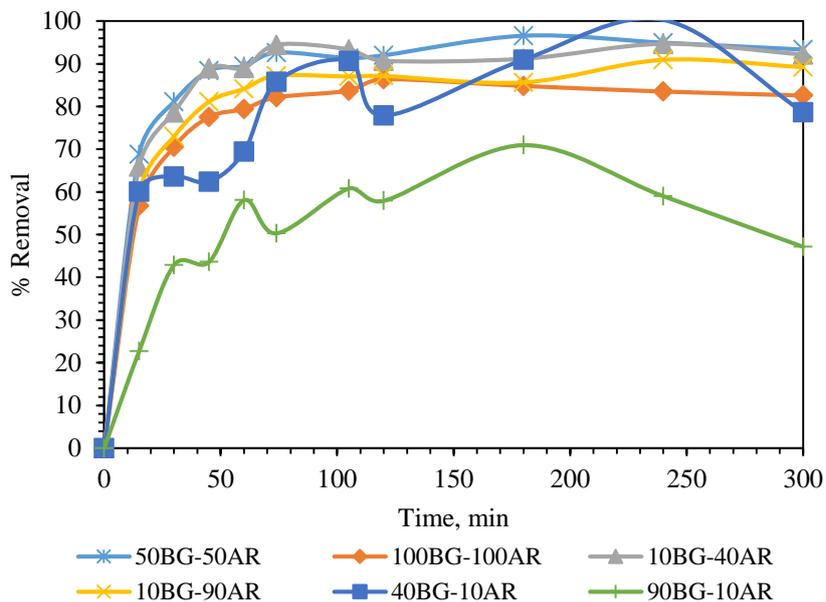


**Figure 5.** ARBG solution at different initial concentration

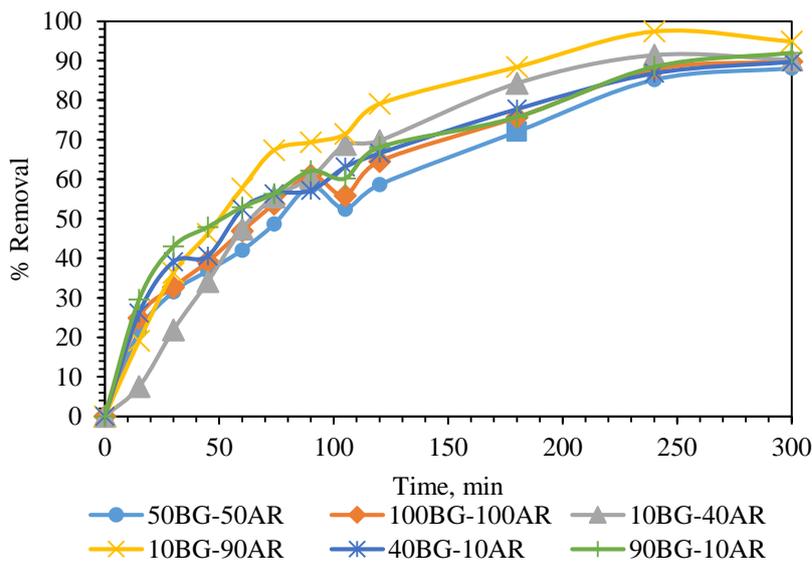
The changes in concentration after 4 hours of adsorption process has been monitored and recorded. The data obtained was used to calculate the percentage removal (%) of the dye concentration in ARBG solution. Figure 6 and Figure 7 shown the percentage removal of initial dyes concentration at 531 nm and 625 nm, respectively. The highest percentage removal of AR1 dye has been recorded at 50BG-50AR (ppm) by having 93.32 % reduction while the lowest was achieved 47.14 % removal at 90BG-10AR (ppm). However, the optimum condition for 40BG-10AR (ppm) to achieve 100% was within 200 min.

As depicted in Figure 7, it shown that the trend of all percentage removal of BG dye at different initial concentration were uptrend and almost similar. There was not much deviation occurred along the adsorption processed within 300 minutes. The percentage removal were kept increased with respect to time as the highest percentage removal was achieved at 94.86 % for 10BG-90AR (ppm) as the concentration of BG dye was the lowest compared to others. As a conclusion, at lower concentration of both cationic and anionic dyes, the highest removal could be obtained and achieved. The ZwiTAd effectively functioned to remove binary dyes simultaneously. The images of colour changes of dyes solution in cuvettes was shown in

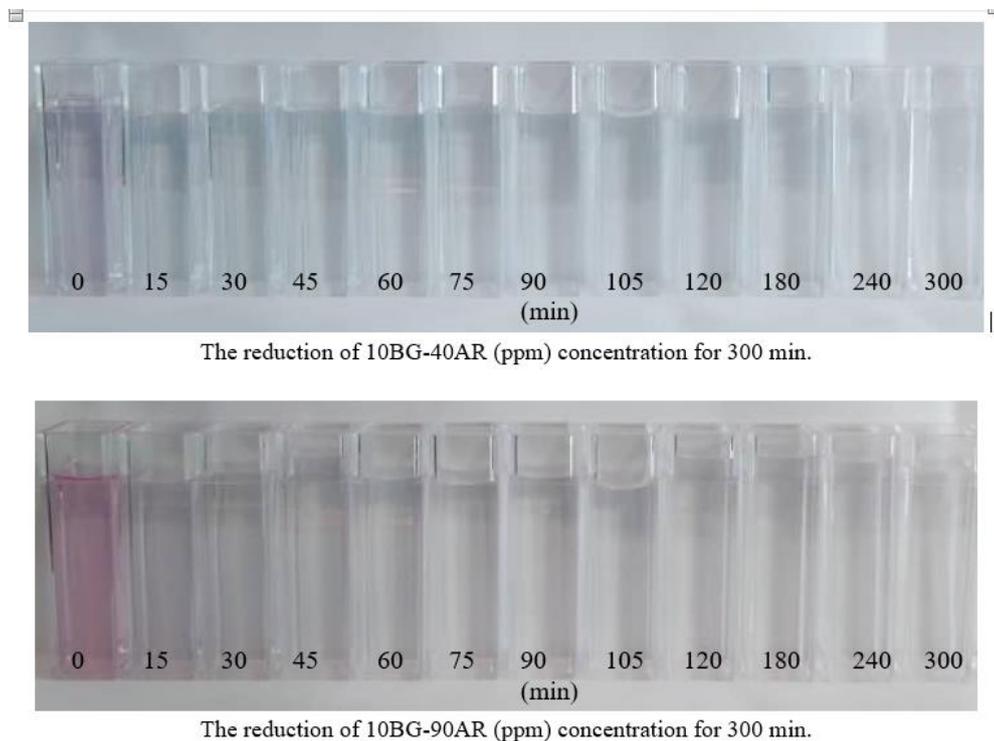
**Figure 8.** The depletion of colour can be seen clearly as time passing from zero to 300 minutes and eventually a clear solution was obtained.



**Figure 6.** Percentage removal at different initial dyes at  $\lambda_{max}$  531 nm



**Figure 7.** Percentage removal at different initial dyes at  $\lambda_{max}$  625 nm

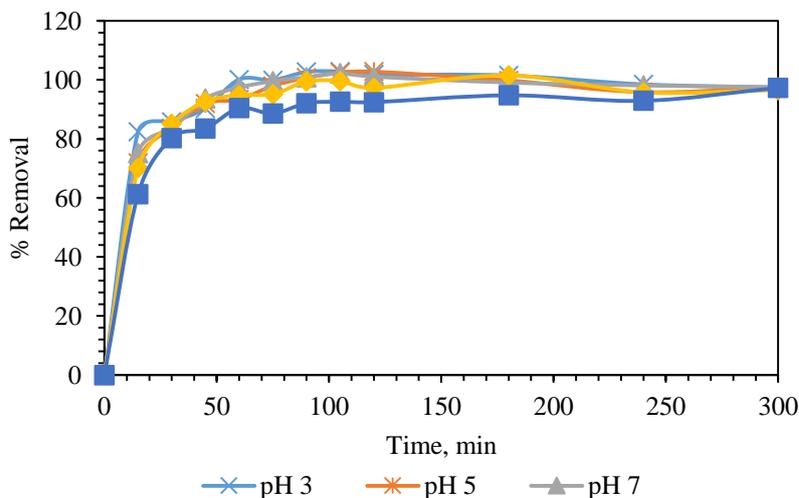


**Figure 8.** The images of colour changes of dyes solution in cuvettes for 300 minutes.

### 3.3. Effect of Solution pH

The pH of dye solution plays an important role in the entire adsorption process, especially as a regards to the adsorption ability [20]. [Figure 9](#) shows the percentage removal of the AR1 at different pH values. The graft shown similar trend as there was not much deviation throughout the experimental process till 300 min. The highest percentage removal at 50BG-50AR (ppm) for AR1 was achieved around 97% for all pH series. This result shown that the anionic dyes in 50BG-50AR (ppm) able to be removed effectively at any range of pH compared to cationic dyes. However, the ZwitAd was dominant towards the removal of anionic dye.

As for the BG dye, the fastest removal was shown at pH 3 where within 50 min, the adsorption have achieved 60% reduction and the slowest removal was at pH 9 ([Figure 10](#)). However, after 300 minutes of adsorption processed, the removal of BG dyes achieved approximately 90% to 93% ranging from acidic to alkaline of pH. This proved efficiency of ZwitAd to remove binary dyes. [Figure 11](#) shown the summary of the percentage removal of AR1 and BG at 50BG-50AR (ppm) dyes.



**Figure 9.** Percentage removal of Acid Red 1 in 50BG-50AR (ppm) at various pH

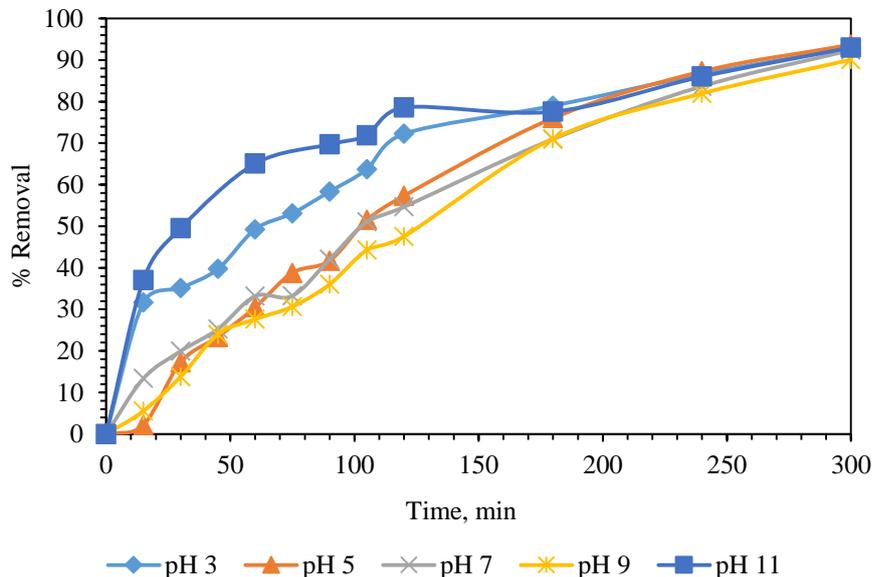


Figure 10. Percentage removal of brilliant green in 50BG-50AR (ppm) at various pH

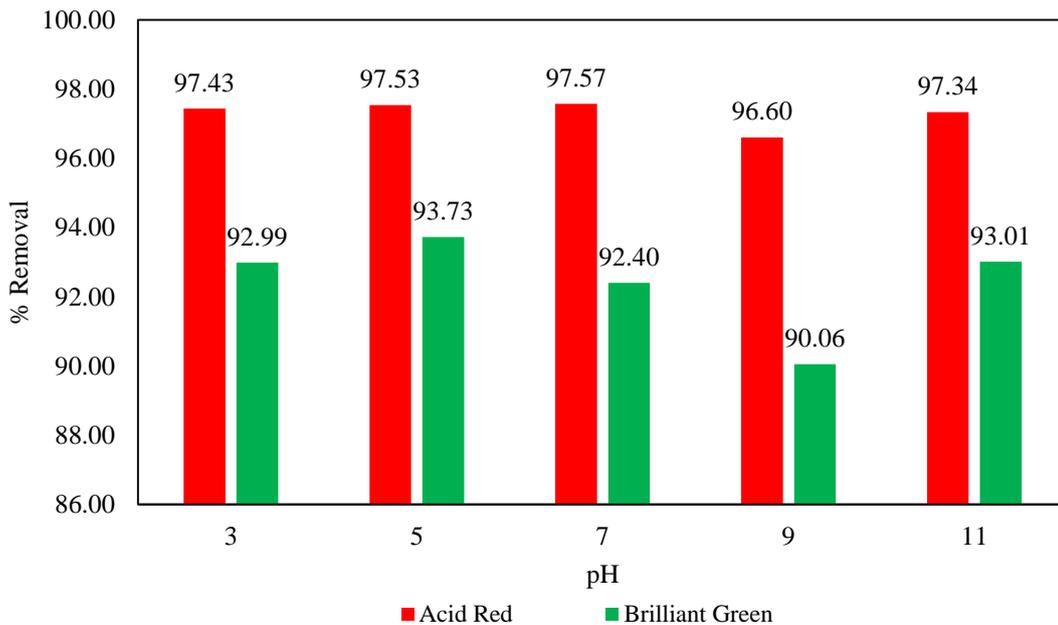


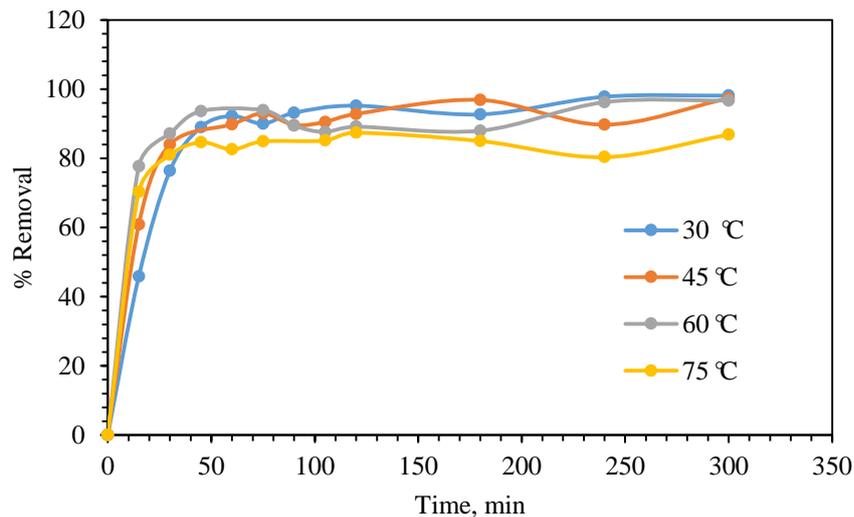
Figure 11. Study effect of pH at 50BG-50AR ppm binary dyes.

### 3.4. Effect of Solution Temperature

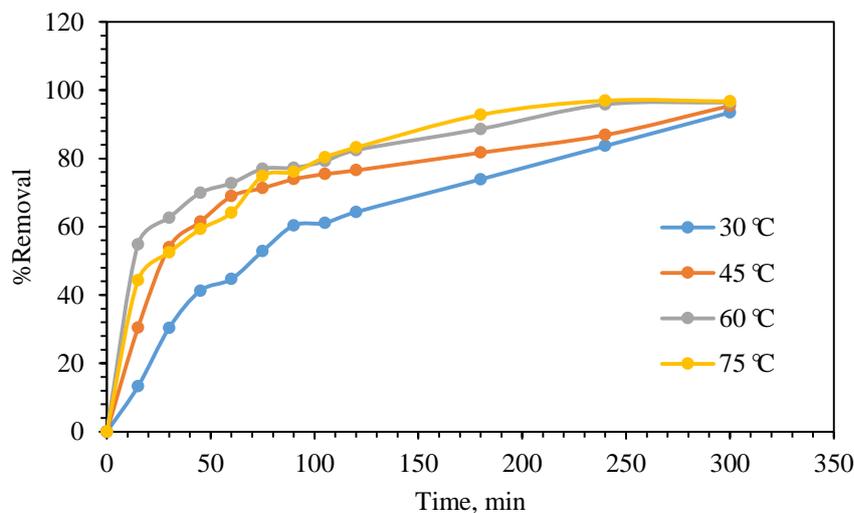
The effect was investigated at different solution temperatures where, 30, 45, 60 and 75 °C were taken into consideration to demonstrate the effect of temperature on ARBG solution. Percentage removal with constant initial concentrations was conducted for this research at 50BG-50AR (ppm). Figure 12 shows the percentage removal of AR1 at 50BG-50AR (ppm). The highest percentage of removal was stated to be at 30 °C which represents at 98.14 % and the lowest percentage of removal was at 75 °C. In the case of anionic AR1, adsorption ability decreased marginally with rising temperature, which indicates that the interactions between the anionic dye and ZwitAd's active adsorption site were lower at the higher temperature. The increased temperature facilitated the mobility of dye molecules and resulted in pigment molecules escaping from the solid to the liquid phase. Moreover, with high temperature, this could lead to the breakdown of existing intermolecular hydrogen

bonding between dye and ZwitAd which is an important contribution to any adsorption process [21]. Thus, might result in increased colour solubility and stronger interaction forces between colorants and solvents than those between colorants and ZwitAd.

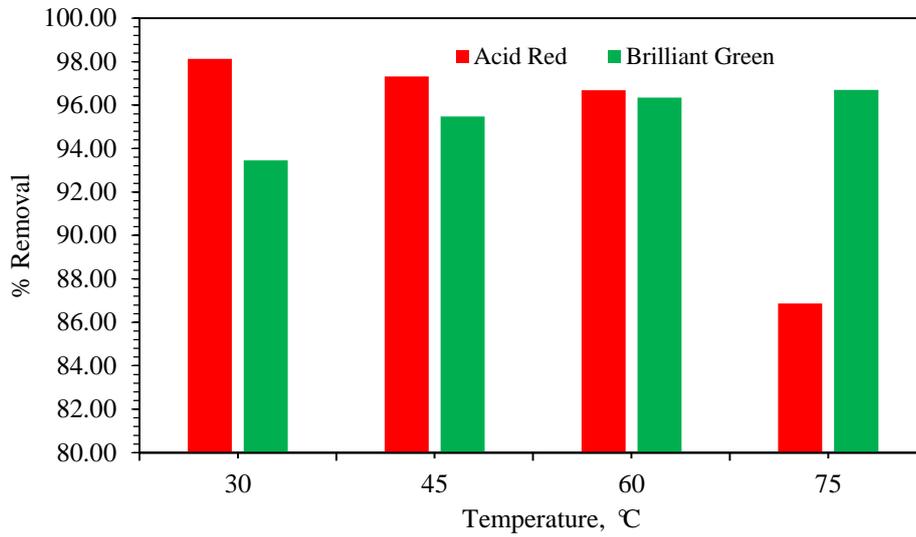
Different case with percentage removal of BG dye where all the temperature condition gives higher removal at 300 minutes as it enhanced the removal by increasing the temperature from 30 °C to 75 °C (Figure 13). That suggests the endothermic existence of ZwitAd's BG adsorption. ZwitAd found the maximum amount of Brilliant Green removed increased from 93.46 % at 30 °C, 95.47 % at 45 °C, 96.35 % at 60 °C followed the highest percentage removal which is 96.70% at 75 °C (Figure 14). This happens because the adsorbent molecule gets enough energy to interact with the adsorbent surface sites. The increased adsorption potential of BG may be due to the enlargement of the pore size, which offers new adsorption sites for coloring interactions that were not possible at lower temperatures. Thus, the rate of intraparticle diffusion of adsorbed molecules into the pores of the adsorbent surfaces was directly increased. Figure 15 displayed an image of the gradient reduction of binary dyes for each of the temperature studied. As can be seen from the images, the binary dyes become clear solution after approximately 300 minutes of adsorption processed carried out.



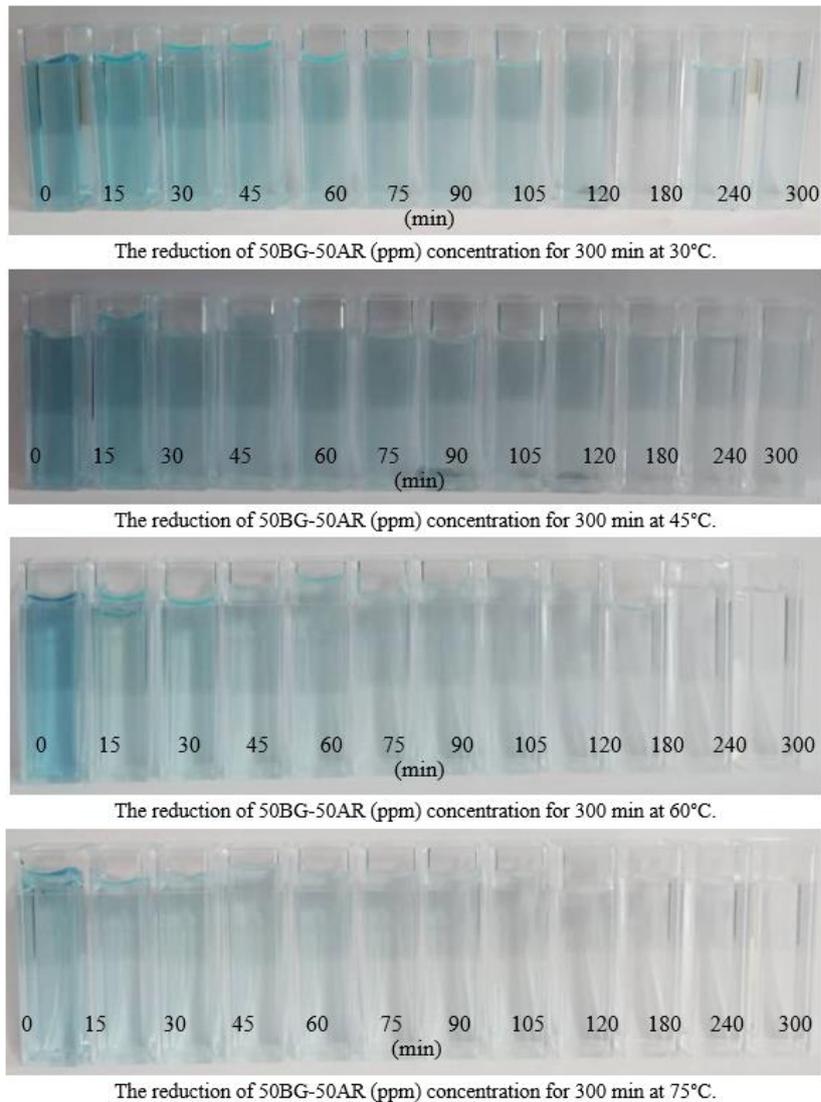
**Figure 12.** Percentage removal of Acid Red 1 in 50BG-50AR (ppm) at various temperature.



**Figure 13.** Percentage removal of Brilliant Green in 50BG-50AR (ppm) at various temperature



**Figure 14.** Percentage removal of 50BG-50AR (ppm) binary dyes at various temperature.



**Figure 15.** The gradient reduction of binary dyes for temperature study effect.

### 3. Conclusion

The adsorption of textile dyes onto zwitterionic adsorbent coating has been done successfully for both single and binary dyes which are involved with the mixture of Acid Red 1 and Brilliant Green. Acid Red 1 is an anionic dye as it represented for direct, acid and reactive dyes. The ability of the adsorbent coating to adsorb Acid Red 1 in ARBG solution is proved as the concentration of dye solution is decreasing. In the same case to Brilliant Green, the concentration before and after using the adsorption process was recorded and it shows that the concentration is reduced. Hence, Zwitterionic adsorbent coating can be used in a binary system as it can adsorb both positive and negative charge of the dyes.

The optimum parameter has achieved to obtain the maximum percentage of the concentration ARBG removal. The higher percentage of removal has been recorded at 92.14% and 90.18% for Acid Red 1 and Brilliant Green respectively. This condition has been used at 10BG-40AR (ppm). Meanwhile, for the initial pH, the best pH to achieve the maximum removal is at pH 11 as both of Acid Red 1 and Brilliant Green is at 100% of the removal. The final would the initial temperature and the optimum condition for this parameter would be at 30 °C as the percentage removal for both dyes at 98.14% and 93.46% respectively.

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