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Original Research Article

Powdered of Highly Porous Hydrogel Nanocomposite Prepared from Acrylamide- Acrylic Acid -Treated by Activated Carbon for Brilliant Blue Dye Removal

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ABSTRACT

With the ex-potentially increase pollutants of dyes, the purification of cationic dye waste water has been an urgent environmental problem. As a new kind of porous adsorbent, hydrogel modified activated carbon frameworks still face challenges in agglomeration, regeneration, reuse ability, and synthesis environmentally un-friendly. In this work, activated carbon was grown *in situ* on the surface of sodium alginate (AAc-AM) beads to fabricate (AAc-co-AM)/AC. This study works to examine the elimination of Brilliant Blue (BB), a harmful and persistent dye that could cause extensive ecological damage, from an aqueous solution via adjusting the amount of acrylamide (AM) and the degree of co-polymer cross-linking. The co-polymer of hydrogels efficiently removes BB in a brief time frame. The synthesized hydrogel was characterized via FESEM and TEM. The best adsorption efficiency of the nanocomposite was 114.4 $mg.g^{-1}$. Reactivation appears to indicate that the material could be utilized repeatedly and has a better ability to resist interference. In addition, the percentage removal of BB dye by hydrogel from aqueous solution were above 91.40%. Therefore, the hydrogel appears great potential for the wastewater.

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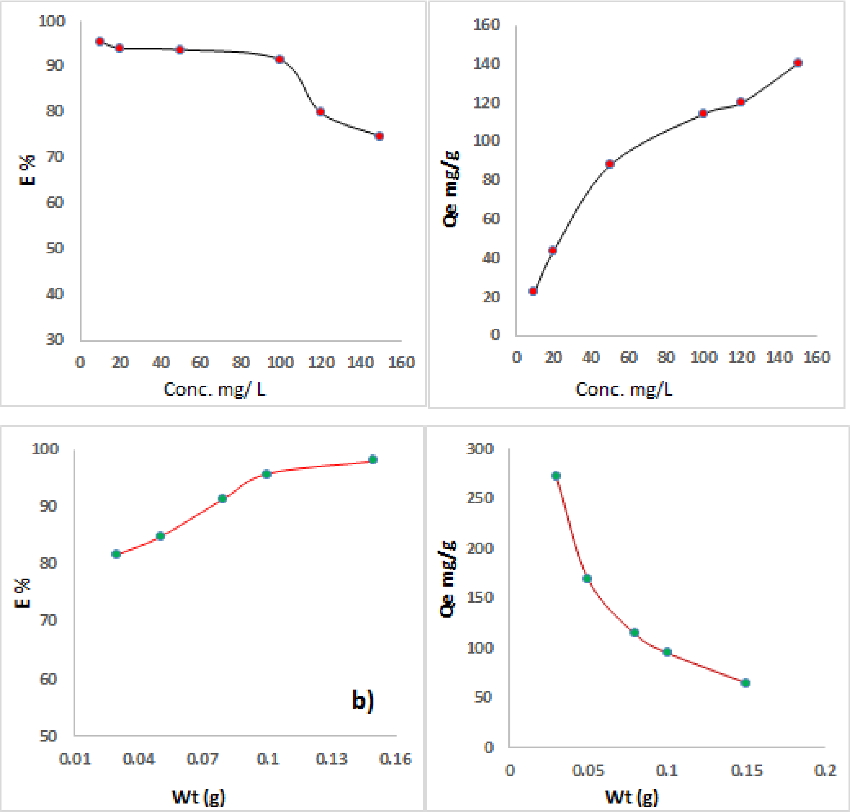
GRAPHICALABSTRACT



Introduction

Pollution of the aquatic environment due to artificial dyes poses a serious concern for all living organisms in the aquatic environment. These toxic pollutants have a clear environmental impact on the ecosystem and pose a major threat to aquatic life [1,2]. Most dyes are toxic and dangerous and have a clear effect on fish and aquatic plants, as well as on human health. Dyes decompose to produce dangerous components that cause genetic mutations and have devastating effects. They also prevent the penetration of light and thus cause disruption to biochemical processes in water bodies. Different methods have been used to treat wastewater contaminated with dyes [3,4]. One of the most important techniques for treating polluted water is the adsorption process because of its economic advantages and the use of highly effective surfaces prepared from less economical materials in treating dyes [5,6].

The dye used in this study, Brilliant Blue (BB), is a common coloring material in the textile, leather, paper, wool, and other industries. It was further used in the past as a common food additive to color dairy products and sweets, but because of its dangerous and toxic effects on humans and animals, it was banned [2,7-9]. Various chemical, physical, and biological based techniques have been developed to eliminate pollutant (dyes). For instance, membrane bioreactors, membrane filtration, filtration, ion exchange photocatalytic degradation, chemical precipitation coagulation-flocculation, and adsorption are useful to eliminate dyes from water. Among these, adsorption has been a preferred technique in the decontamination of dye waste water since it is both highly efficient and ecofriendly with no secondary contamination [10,11].

Activated carbon is a commonly used term used to describe carbon-based materials that have a large surface area and high pore structure. Large quantities of alternating current are produced from various carbon-rich materials such as coal, fruit peels, and wood [12]. The advanced porosity and large surface area, which consists of several pores, in addition to contain wide active aggregates with an effective surface function, has important and wide uses that make it a material with multiple uses and has many applications in different fields, but mainly in the environmental field [13-15] .

Hydrogels consist of a three-dimensional network of several monomers, usually natural polymers and synthetic polymers. Examples of natural polymers are polysaccharides such as sodium alginate and carboxymethyl cellulose, starch, chitosan, and others, while the synthetic polymers are acrylic, acrylamide, and others. [16]. Recently, natural hydrogels have been replaced by synthetic hydrogels due to their high ability to swell and insoluble in water, and also have a lower economic cost and can be regenerated and used [17].

Therefore, in this study, a new hydrogel based of activated carbon (AC) was prepared by copolymerization method as higher absorbents to removal dye from aqueous solution. The synthesized hydrogel was characterized via FTIR, FESEM, and TEM. Furthermore, the adsorption efficiency and removal percentage of dye was obtained by optimization of adsorption parameters.

Experimental

Preparation of standard solution

A 500 mg/L BB dye is prepared via adding 0.5 g in 500 mL distilled water. Serial dilutions are prepared for the experimental methodology. UV-Vis spectrophotometer was used for detecting the concentration of dye at 590 nm, Calibration curves were plotted among absorbance and concentration of dye solution. Equation 1 is used for calculating the removal percentage, while Equation 2 is used for determining the adsorption capacity.

$E\% = (Co - Ce)/Co \times 100$ (1)

$Qe = (Co - Ce)\ V/M$ (2)

Where, Ce mg/L indicates equilibrium concentration and Co mg/L describes initial.

Preparation of (AAc-co-AM)/AC hydrogel

Acrylic amide (AM) grafted with acrylic acid (AAc) was prepared by loading activated carbon (AC) by free radical graft co-polymerization mechanism in the presence of initiator KPS. In the reaction flask, a weight of activated carbon (0.1 g) was taken in 10 mL of distilled water and shaken for 30 min. After that, 1 g of AM was dissolved in 10 mL distilled water and the reaction mixture stirring. 5 mL of monomer (AAc) and 0.008 g in 2 mL of MBA were added to the reaction mixture followed by dropwise addition of initiator KPS (0.003 g) in 2 mL deionized water. Nitrogen gas purge was used throughout the process. Free radical co-polymerization was performed at 80 °C. Finally, the resulting product was dried in an oven at 60 °C.

Result and Discussion

FT-IR of the hydrogels in Figure 1a depicts the (AAc-co-AM)/AC hydrogel before adsorption, while Figure 1b shows the (AAc-co-AM)/AC hydrogel after adsorption. The FT-IR spectra of (AAc-co-AM)/AC hydrogel revealed the presence of characteristic utilitarian aggregates in the hydrogel. The broad upper part at 3410.77 cm^{-1} was attributed to the O–H stretching vibration and the C–H vibration appeared at 2934.13 cm^{-1}. The band seen at 1422.41 cm^{-1} relates to O–H bending or C–H torsion regions for a stretching vibration at 1233.87 cm^{-1}. The low impact band at 541.32 cm^{-1} was associated with C–O or O–H (out-of-plane) bending vibration [18]. The band at 1730.92 cm^{-1} is attributed to the presence of an amide functional group in hydrogel. Figure 1b of the hydrogel after the adsorption process, that no change in the composition of the surface of the preparation or the appearance of a new beck, only a displacement in the beck. This is evidence of the occurrence of physical adsorption and the success of the adsorption process [19,20].

Figure 2 displays FESEM images of (a) (AAc-co-AM)/AC before adsorption and (b) (AAc-co-AM)/AC after adsorption. The FESEM images of before adsorption revealed that it had an uneven form, a rough surface, and pores. FESEM images after the adsorption process indicated that the irregular shape of the hydrogel surface had disappeared and the appearance of small rough particles of different shapes and with an average size of 100 nm were seen on the hydrogel surface. The appearance of small rough particles observed within the (AAc-co-AM)/AC hydrogel polymer chain clearly indicated that they resulted from the grafting process of the AAc-co-AM hydrogel with activated carbon [21,22].

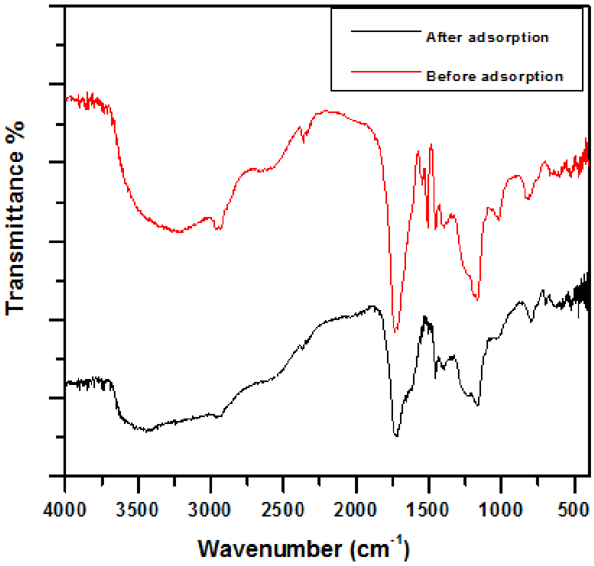


Figure 1. a) FT-IR spectra of (AAc-co-AM)/AC before adsorption and b) (AAc-co-AM)/AC after adsorption presses.

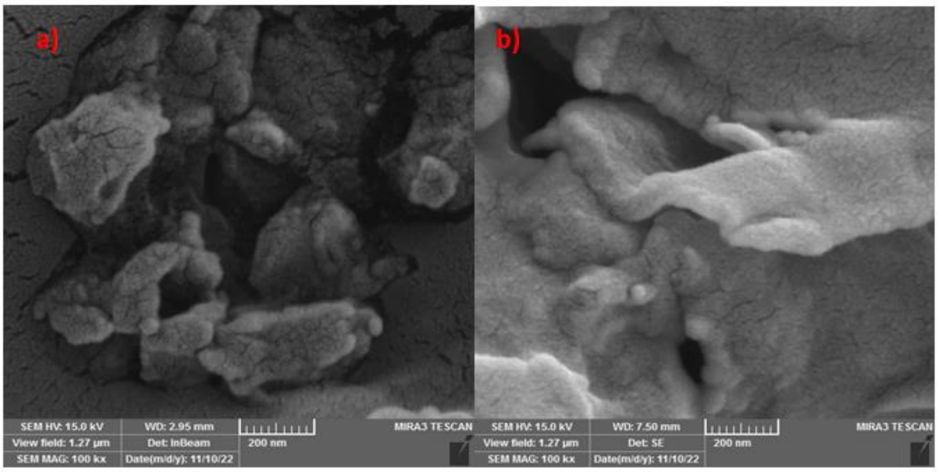


Figure 2. FESEM image a) (AAc-co-AM)/AC before adsorption and b) (AAc-co-AM)/AC after adsorption.

The morphological characterization of the AAc-co-AM hydrogel and (AAc-co-AM)/AC hydrogel was performed by TEM in Figure 3 that illustrates the morphology of compounds as nanoparticles is a rectangle or cube. The vast majority of hydrogel had sizes ranging from 25-50 nm. It was observed in Figure 3a smooth surface devoid of cavities, while in Figure 3b, after loading the activated carbon on the hydrogel, the figure began to contain dark spots, evidence of loading the activated carbon [23-25].

Adsorption of (AAc-co-AM)/AC hydrogel

It is very significant to study the influence of several reaction factors when studying the adsorption efficiency of complete adsorbents. (AAc-co-AM)/AC weight of hydrogel, concentration, and solution temperature, and pH shown in Figure 4a–d. The effect of many factors like mass of hydrogel (0.03–0.15 g/L), concentration of dye (10-150 mg/L), and solution temperature (15-40 °C), pH=3-11.

The concentration of dye about of 10-100 mg/L at 30 °C with mass of (AAc-co-AM)/AC hydrogel (0.08 g) of for 60 min. Figure 4a has the best adsorption at 100 mg/L of dye. Furthermore to that increase concentration of dye from (10-150 mg/L) decreases removal percentage% from (74.44 % - 95.87 %) that could likely be due to the improving in the number of molecule of dye. But increase concentration of dye increase adsorption capacity [26,27].

The considerable parameter is weight of hydrogel which effect the presence of number of active site on the adsorbent surface. The results of (AAc-co-AM)/AC hydrogels ranged (0.03-0.15 g/L) and the outcome showed in Figure 4b have generalized that best weight of hydrogel uptake at (0.08 g) equilibrium before dye and hydrogel was adopted at these mass and primary rises can be ascribed to the increases in concentration of active site for adsorption. Adsorption capacity decreases with increase weight of hydrogel from (65.55 -262.55 mg/L), while removal percentage increase from (81.11%-98.88 %). Additional rise did not alter the removal percentage as the necessary active site was carried at 0.08 g/100 ml dose for a specific concentration [24].

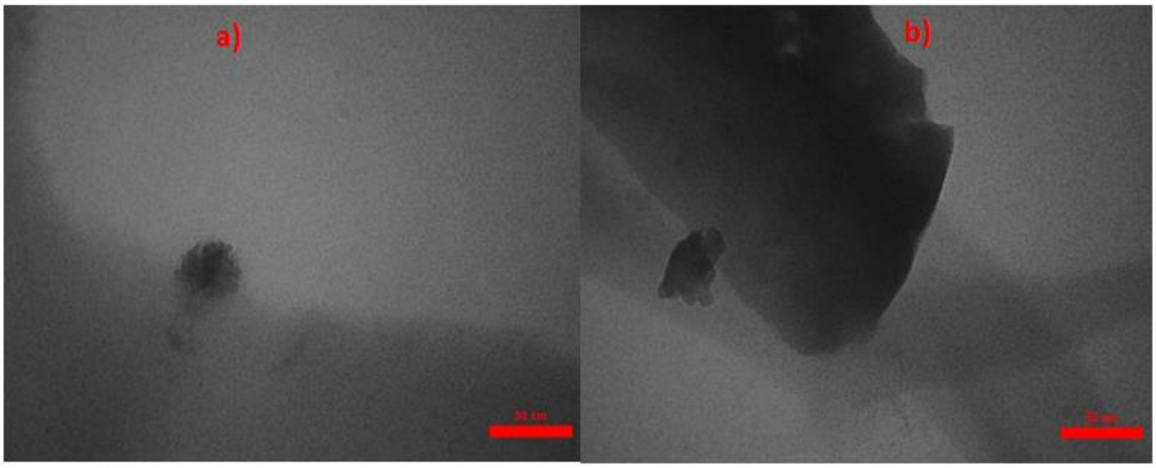


Figure 3. TEM image of morphological characterization of the a) AAc-co-AM and b) (AAc-co-AM)/AC.

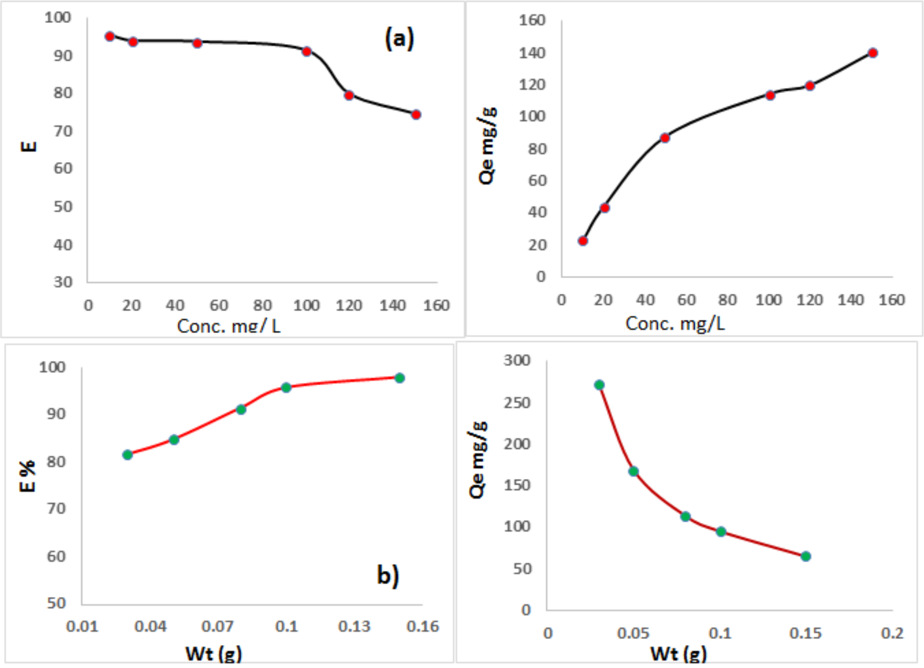


Figure 4. Optimization of adsorption parameters (a) concentration of BB dye and (b) weight of (AAc-co-AM)/AC hydrogels.

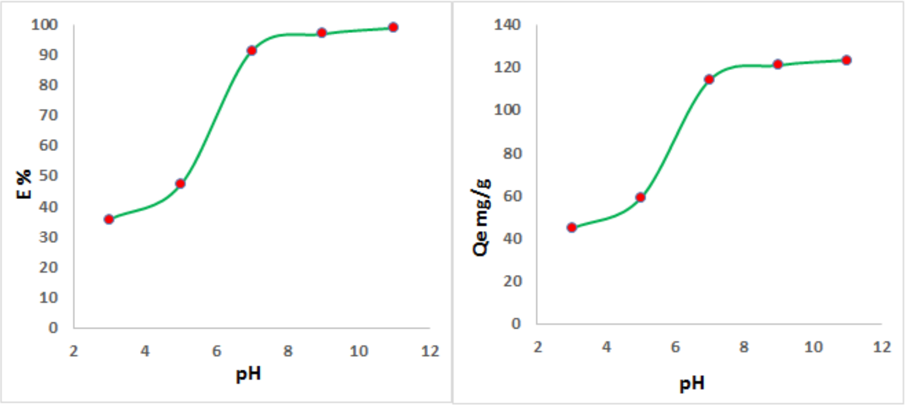


Figure 5. Effect of pH solution onto removal efficiency of BB dye by (AAc-co-AM)/AC hydrogel.

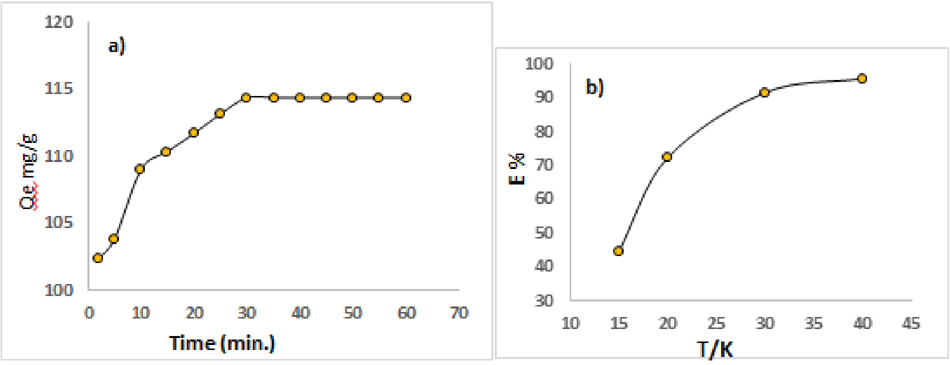


Figure 6. Optimization of adsorption parameters (a) equilibrium time, (b) temperature of BB dye onto (AAc-co-AM)/AC hydrogels.

The pH is essential factors in adsorption method which not only demonstrated altering efficiency of ions among adsorbate molecule and adsorbent, however too influence properties the adsorbent surface in addition to the surface of adsorbate, these to entrapment diametrically in different solution pH. The influence of pH on adsorption method was appraised at diverse pH level (Figure 5) [28]. Results found the percentage removal of dye by (AAc-co-AM)/AC hydrogel was raised as solution pH was raise from pH=3-10. 35.99 % at pH=3 which increase to pH 7 about 91.88 % which can be credited to the protonation of (AAc-co-AM)/AC hydrogel. Furthermore, rise in pH dropt of the removal percentage pH 10 at 98.41% [29,30]. The influence of equilibrium time of this hydrogel with dye limits the best optimum time necessary for maximum equilibrium time. The adsorption of dye carried out in the selected series of (5-60 min) with 100 mg/L initial dye concentration with mass of hydrogel 0.08 g as appear in Figure 6a. The best adsorption capacity is found at 60 min and thereafter equilibrium attainment best removal percentage 81.45%. With further rise in time, 91.39% adsorption remained almost constant possibility due to obtainability of greater active site. Thus, equilibrium time selected for subsequent experiment was 60 min [31,32].

The solution temperature ranges about 15-40 °C was tried for the study, and the results are displayed in Figure 6b, where the greatest removal percentage % from 44.44 % to 95.47% was reached at 30 °C. At this temperature, no species are normally needed for the interaction of the molecule dye with the AAc-co-AM)/AC. In contrast, the reaction can be defined as endothermic process [33].

Conclusion

The role of loading activated carbon on the surface of sodium alginate (AAc-AM) beads was to fabricate (AAc-co-AM)/AC. This study elimination Brilliant Blue (BB), a harmful and persistent pigment that can cause widespread ecological damage, from an aqueous solution via adjusting the amount of acrylamide (AM) and the degree of cross-linking of the co-polymer. Increase concentration of dye from (10-150 mg/L) decreases removal percentage% from (95.55-74.44 %), and adsorption capacity decrease with increase weight of hydrogel from (65.55 -262.55 mg/L), while removal percentage increase from (81.11%-98.88 %). Additional rise did not alter the removal percentage as the necessary active site was carried at 0.08 g/100 mL dose for a particular concentration. Results found the percentage removal of dye by (AAc-co-AM)/AC hydrogel was raised as solution pH was greater than before from pH=3-11. 35.82% at pH=2 which increase to pH 7 about 91.41% which can be credited to the protonation of (AAc-co-AM)/AC hydrogel. Furthermore, rise in pH dropt of the removal percentage pH 10 at 98.41%.

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