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CONFERENCE PAPER

Adsorption of Remazol Brilliant Blue R by raw and carbonized macroalgal wastes

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ABSTRACT

In this study, adsorption of Remazol Brilliant Blue R (RBBR) dye was carried out using *Ulva lactuca*, *Codium fragile* and their carbonization products; adsorption kinetics and isotherms were determined. Carbonized adsorbents were produced in a tubular furnace at the heating rate of 20°C min^{-1} and temperature of 500 °C with a nitrogen flow rate of 300 ml min^{-1}. Adsorption was carried out in a shaking water bath at the temperature of 25 °C and shaking speed of 200 rpm for 2 h. Carbonized adsorbents have been found to have greater adsorption capacity than raw biomass. It was seen that the adsorption lasted for 90 and 60 minutes for raw and carbonized adsorbents, respectively and then desorption begun. Maximum adsorption was achieved with the carbonized *Ulva lactuca* macroalgae (2.11 mg g^{-1}). It has been found that the most suitable kinetic model for all absorbents was the pseudo-second-order kinetic model. The best fit of equilibrium data for all adsorbents was described by the Langmuir model.

Keywords: Adsorption, carbonization, macroalgae, remazol brilliant blue R

1. INTRODUCTION

Water pollution is one of the most important problems which society is facing today. Recent environmental problems and the development of the industry have led to a gradual decline in the amount of potable water [1]. Especially the synthetic dyes used in the industry cause serious water pollution. Therefore, there are many studies in the literature regarding the removal of dyes from industrial wastewaters [2]. Today, there are many physical and chemical methods such as adsorption, chemical coagulation, precipitation, ultra-filtration and ionizing radiation which are effectively used to remove synthetic dyes. Among these methods, adsorption is considered as a superior technique comparatively with other techniques due to the availability of many adsorbents, easy design and operation and high efficiency [3-6].

The adsorption process takes place due to presence of unbalanced or residual forces at the solid surface that is termed the adsorbent. Adsorption is affected by the solid surface and dissolved gas or solute in the solvent. Adsorption is essentially a surface phenomenon. Adsorption process involves two components: Adsorbent and adsorbate. Adsorbent is the substance on the surface of which adsorption takes place. Adsorbate is the substance which is being adsorbed on the surface of adsorbent [7].

In general, adsorbents should have a high surface area for more efficient adsorption. Depending on the application, the surface may be hydrophobic or hydrophilic [7]. Different adsorbents such as carbon nanotubes (CNTs) have been conventionally used for the adsorption process, whereas activated carbon has been widely used in the recent past [2]. Activated carbon has been studied for the adsorption process not only at laboratory scales but also for the industrial applications and it has been seen that the obtained results are quite remarkable [8],[9]. However, usage of activated carbon is limited due to relatively expensive material and operation costs. Thus, researchers are studying to produce low-cost carbon-based adsorbents from alternative feedstocks such as agricultural wastes and algal wastes [2, 10].

This work focused on evaluation of the potential of different algal adsorbents (*Ulva lactuca*, *Codium fragile*, carbonized *U. lactuca* and carbonized *C. fragile*) for the removal of Remazol Brilliant Blue R

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(RBBR) from aqueous solution. The adsorbents were characterized by Fourier transform infrared spectroscopy (FT-IR) and proximate analysis. In the batch adsorption experiments, adsorption kinetic data were determined, pseudo-first-order and pseudo-second-order kinetic models were tested to fit these adsorption kinetic data. The equilibrium data were analyzed using Langmuir and Freundlich isotherm models.

2. MATERIALS AND METHOD

2.1 *Preparation and Characterization of the Adsorbents*

Ulva lactuca, Codium fragile and their carbonized products were used as adsorbent in this study. Macroalgae samples were collected from coastal areas of Marmara Sea and washed with distilled water to remove impurities such as shell and sand. These algae were dried at 70°C for a night and then powdered using a grinder. The obtained *Ulva lactuca* and *Codium fragile* were referred as UL and CF, respectively.

The production of carbonized adsorbents were conducted using the procedure described by Kocer et al. [11]. The carbonization process was performed in Protherm model split typed furnace. Obtained UL and CF powder were carbonized at the temperature of 500 ^{0}C, heating rate of 20°C min^{-1}, retention time of 30 min and with nitrogen flow rate of 300 mL min^{-1}. Before the experiments, inside of the furnace were swept with nitrogen gas for 15 minutes to provide an inert atmosphere. After the carbonization process, the furnace was cooled and the samples were placed in a desiccator for characterization and adsorption experiments. The obtained carbonized *Ulva lactuca* and *Codium fragile* were referred as CUL and CCF, respectively.

Proximate analyzes of the UL and CF were carried out using the thermogravimetric analyzer (TA Instrument, SDT Q600). In proximate analysis, moisture content, volatile matter content, fixed carbon content and ash content were determined according to Ozcimen [12]. The functional groups present at the surface of the UL, CF, CUL and CCF were identified by FT-IR in the range of 4000–600 cm^{-1} using a Thermoscientific Nicolet 6700 spectrophotometer.

2.2 *Adsorption Experiments*

The adsorption experiments were performed using the procedure described by Ozcimen and Salan [2]. In these experiments, UL, CF, CUL and CCF were used to remove Remazol Brilliant Blue R (RBBR) dye from aqueous solutions. Firstly, 1000 mg L^{-1} stock solution of RBBR was prepared and then this stock solution was diluted to standard concentrations of 20, 40, 60, 80 and 100 mg L^{-1}. The pH values of the standard solutions were adjusted to 3 in all cases with HCl/NaOH solutions due to provide increased interactions between the adsorbent and reactive azo dyes. At lower pH, the reactive azo dyes such as anthraquinonic RBBR dissolves and releases colored negatively charged dye anions into aqueous solution, which will exhibit electrostatic attraction towards positively charged surfaces. Moreover, at acidic pH values, some functional groups of adsorbent are also protonated. The dissociated anions of dye molecules are transferred from solution to the surface of adsorbent and adsorption occurs via the electrostatic interactions between the ions of negatively charged dye molecules and positively charged absorbent surface [2].

In the batch adsorption experiments, 40 mg adsorbent was thoroughly mixed with the 5 ml aqueous solution of dye (60 mg L^{-1}) in a sealed conical centrifuge tube placed into a tube rack. These experiments were carried out at a constant agitation speed of 200 rpm and temperature of 25 ^{0}C for 120 min. After each adsorption experiment was completed, residual concentration of RBBR was determined by spectrophotometric analysis (PG Instruments T60 UV-Visible Spectrophotometer) in the visible range at the wavelength of 593 nm (λ_{max}). The amount of the remained RBBR was determined from the calibration curve obtained according to concentration-absorbance chart of initial standard solutions. The adsorbed quantity was calculated using the following equation:

$$Q = \frac{(C_0 - C_e).V}{W} \tag{1}$$

where C_0 and C_e (mg L^{-1}) are the amount of initial and remaining RBBR in the solution at time of equilibrium respectively, V is the volume (L) of the solution, and W is the weight (g) of the adsorbent.

The kinetic data were fit by employing the pseudo-first-order [13] and pseudo-second-order [14] models, as expressed by Eq. (2) and Eq. (3) respectively:

Pseudo-first-order equation:

$$\ln(Q_e - Q_t) = \ln Q_e - (k_1.t) \tag{2}$$

Pseudo-second-order equation:

$$\frac{t}{Q_t} = \left(\frac{1}{Q_e^2.k_2}\right) + \frac{t}{Q_e} \tag{3}$$

where Q_e and Q_t are the amounts (mg g^{-1}) of RBBR adsorbed onto the adsorbents at the equilibrium and at the time of t, respectively, while k_1 and k_2 are the kinetic rate constants for the pseudo-first-order (1 min^{-1}) and the pseudo-second-order (g mg^{-1} min^{-1}) adsorption processes, respectively.

After determination of adsorption kinetics, the experimental data obtained for the equilibrium adsorption of RBBR onto the adsorbents were analyzed employing the Freundlich [15] and Langmuir [16] isotherm with Eq (4) and Eq (5) given below:

Freundlich isotherm equation:

$$\log Q_e = \frac{\log C_e}{n} + \log K_f \tag{4}$$

Langmuir isotherm equation:

$$\frac{1}{Q_e} = \left[\frac{1}{Q_{max}.b}\right].\frac{1}{C_e} + \frac{1}{Q_{max}} \tag{5}$$

where Q_e (mg g^{-1}) and C_e (mg L^{-1}) are the equilibrium concentrations of RBBR dye in the solid and liquid phases, respectively, while K_f [(mg g^{-1})/(mg L^{-1})$^{1/n}$]

and *n* are the Freundlich constants related to the adsorption capacity and intensity, respectively. Similarly, Q_{max} (mg g^{-1}) and *b* (L g^{-1}) are the Langmuir constants related to the adsorption capacity.

3. RESULTS & DISCUSSION

3.1 *Characterization of the Adsorbents*

The results of proximate analysis of the adsorbents were shown in Table 1. As it can be seen in Table 1, UL had maximum moisture content (12.29%), on the contrary, CF had minimum moisture content (9.04%). The reason for the high moisture content of all adsorbents was the moisture holding in the environment by these adsorbents. Therefore, adsorbents should be stocked in the desiccator prior to the adsorption process for increasing adsorption efficiency [17]. Due to the high carbohydrate content of macroalgae, the amount of volatile substances of raw macroalgal adsorbents were higher than volatile contents of carbonized adsorbents [18]. When the ash contents of all adsorbents were compared with each other, it was seen that the highest and the lowest value were belonged to CUL and CF, respectively. According to proximate analysis results, the data obtained from this study were in agreement with the literature [19].

Table 1. Proximate analysis of adsorbents

Adsorbents	Moisture Content (%)	Volatile Matter Content (%)	Fixed Carbon Content (%)	Ash Content (%)
UL	12.29	67.37	6.438	13.69
CF	9.04	66.44	12.65	11.87
CUL	10.26	23.22	29.82	36.70
CCF	10.12	22.03	38.70	29.15

The FTIR spectrums of the adsorbents were shown in Fig 1. As can be seen in Fig 1, O- and H- containing functional groups were not presented in CUL and CCF due to the formation of carbonized macroalgae at high temperatures. The absence of these functional groups in carbonized adsorbents indicates the emission of CO_2, CH_4 and H_2 [20]. The FTIR spectra of UL and CF showed a broad band present between 3600 and 3200 cm^{-1} which can be assigned to the O–H stretching groups which were hydroxyl containing compounds such as water. This band was broader in the FTIR spectrum of UL because UL has more water content than CF [21]. In the spectrums of UL and CF, the bands seen at 2950 cm^{-1} indicated symmetric and asymmetric C-H stretching vibrations originating from aliphatic hydrocarbons. Peaks between 1250 and 1300 cm^{-1} also indicated the aliphatic C-N stretching [22].

When the FTIR spectra of adsorbents were compared with each other, it was seen that the high temperature in the carbonization process causes the transition from aliphatic to aromatic structure, aromatic ring formation and H deformation. During the carbonization process, while the OH and CH_3 bonds decreased, the C = C structures increased [23]. Bands between 700-900 cm^{-1} indicated aromatic structure; bands between 1500-1600 cm^{-1} were due to the aromatic C = C vibration [24].

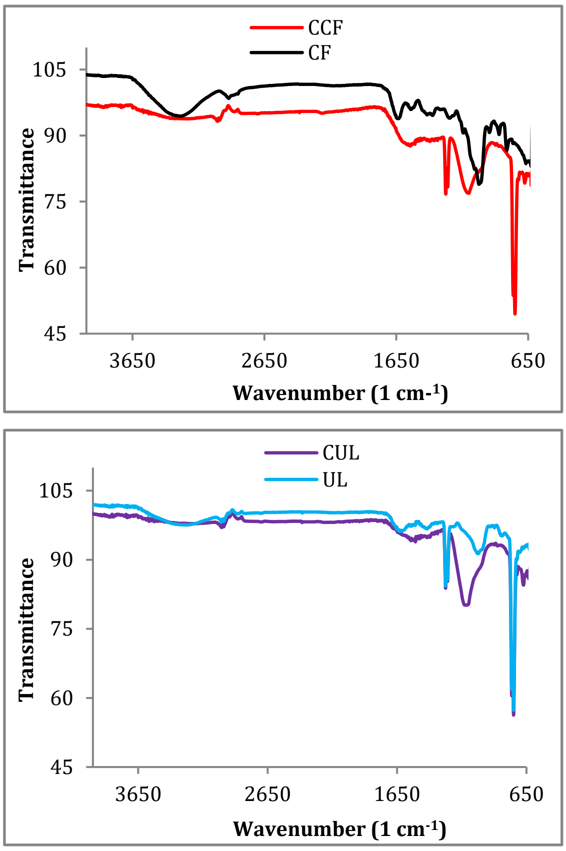

Fig 1. FTIR spectras of adsorbents

3.2 *Adsorption Kinetics*

In order to determine the optimum adsorption time, the effect of contact time on adsorption was evaluated at 25˚C by using 60 mg L^{-1} dye solution and obtained results were shown in Fig 2. According to the Fig 2, the adsorption rate increased rapidly at the beginning of the process and it becomes slow with the increase in the contact time until equilibrium was reached. Furthermore, since the adsorbents were saturated with RBBR, the desorption process started after the equilibrium time [2, 25, 26]. The minimum contact times required for the equilibrium were found as 60 and 90 min for carbonized macroalgae and raw macroalgae, respectively.

As can be seen in Fig 2, the adsorption capacity of carbonized macroalgae was about two times higher than the raw macroalgae. This may be due to network pore structure of carbonized macroalgae and other factor that is generated during the carbonization process which exhibit high specific surface area [27]. CUL showed the best adsorption performance while CF showed worst performance among the four samples.

Pseudo-first-order kinetic model and Pseudo-second-order kinetic model have been investigated to determine what kind of mechanism was carried out during adsorption. Parameters of the pseudo-first-order and pseudo-second-order models were calculated from Fig 2 and the obtained data and the correlation coefficients were given in Table 2.

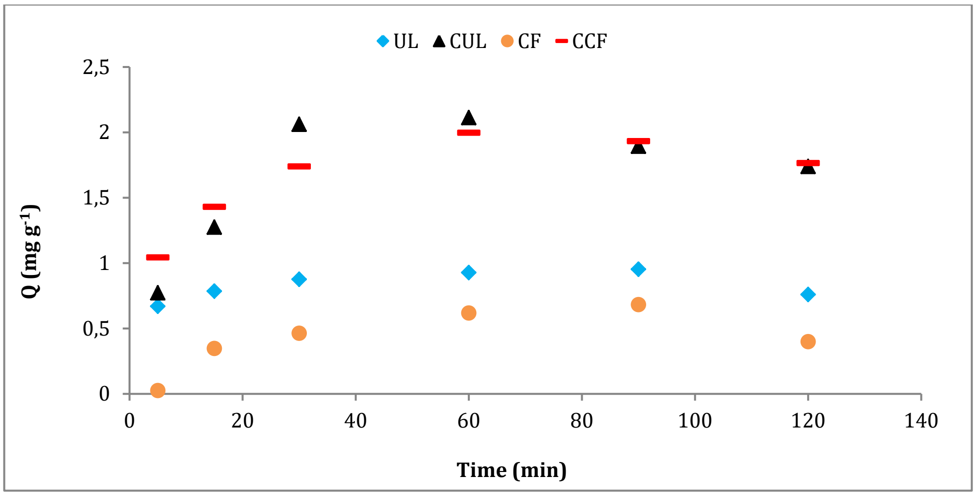

Fig 2. Adsorption capacities of RBBR onto the adsorbents

Table 2. Pseudo-first-order and Pseudo-second-order kinetic models

Adsorbent	Pseudo-First-Order				Pseudo-Second-Order		
	Q_{exp} (mg g^{-1})	k_1	Q_e (mg g^{-1})	R^2	k_2	Q_e (mg g^{-1})	R^2
UL	0.953	0.043	0.324	0.991	0.032	0.983	0.999
CF	0.683	0.023	0.446	0.871	0.049	0.859	0.998
CUL	2.113	0.013	3.652	0.926	0.075	2.129	0.979
CCF	1.997	0.030	0.885	0.959	0.087	2.084	0.997

As can be seen in Table 2, although the correlation coefficient was quite high, Q_e values of adsorbents from pseudo-first order kinetic model were not in agreement with experimental data. Thus, the adsorption process did not comply with this model. The values of Q_e were in agreement with the experimental data (Q_{exp}) for the pseudo-second order kinetic model. The correlation coefficients of pseudo-second-order kinetic model were also higher than that of the pseudo-first order kinetic model. Consequently, the adsorption can be estimated more appropriately by the pseudo-second-order kinetic model for all adsorbents [1, 2, 26].

3.3 Adsorption isotherms

Adsorption isotherms are important for the design of adsorption process because they show how the adsorbates are partitioned between the liquid and solid phases when the adsorption process reaches equilibrium conditions [2, 28]. The above cited constants were determined by nonlinear regression analysis and obtained values and correlation coefficients were given in Table 3.

As it can be seen in Table 3, the best correlation for all adsorbents was obtained with the Langmuir model. Q_{max} values of carbonized macroalgae indicated that the adsorption capacity was found higher than that of raw macroalgae. Q_{max} values were 1.748, 1.417, 3.749 and 2.561 mg g^{-1} for UL, CF, CUL and CCF, respectively. The results indicated that the carbonization process increases the adsorption of dyes. The adsorption performance of RBBR increased approximate two times with carbonization process of macroalgae.

Since, this system is explained with the Langmuir isotherm, the determination of the equilibrium parameter (R_L) is very significant [29]. The equilibrium parameter (R_L) can be calculated by the following equation:

$$R_L = \frac{1}{1+bC_0} \qquad (6)$$

where b (mg L^{-1}) is the Langmuir constant and C_0 (mg L^{-1}) is the initial dye concentration. The value of R_L shows the kind of the Langmuir isotherm to be unfavorable ($R_L > 1$), linear ($R_L = 1$), favorable ($0 < R_L < 1$) or irreversible ($R_L = 0$). Fig 3 presents the R_L values of the adsorption process at different initial dye concentrations for all adsorbents. As it can be seen in Fig 3, all of R_L values were between 0 and 1. This result was shown that the adsorption behaviors of RBBR dye onto all adsorbents were significantly favorable [2]. Table 4 shows a summary of related studies about RBBR adsorption onto different adsorbents and their adsorption performance comparing the results of this study. Considering the previous studies about the RBBR uptake by adsorbents obtained from various sources, macroalgae samples and carbonized macroalgae underperform in RBBR dye removal.

Table 3. Freundlich and Langmuir isotherms constants

Adsorbent	Freundlich			Langmuir		
	n	K_f (mg g^{-1})/(mg L^{-1})$^{1/n}$	R^2	Qmax (mg g^{-1})	b (L g^{-1})	R^2
UL	2.834	0.338	0.849	1.748	0.112	0.940
CF	2.341	0.163	0.958	1.417	0.031	0.979
CUL	1.121	0.026	0.964	3.749	0.007	0.981
CCF	1.839	0.198	0.878	2.561	0.052	0.899

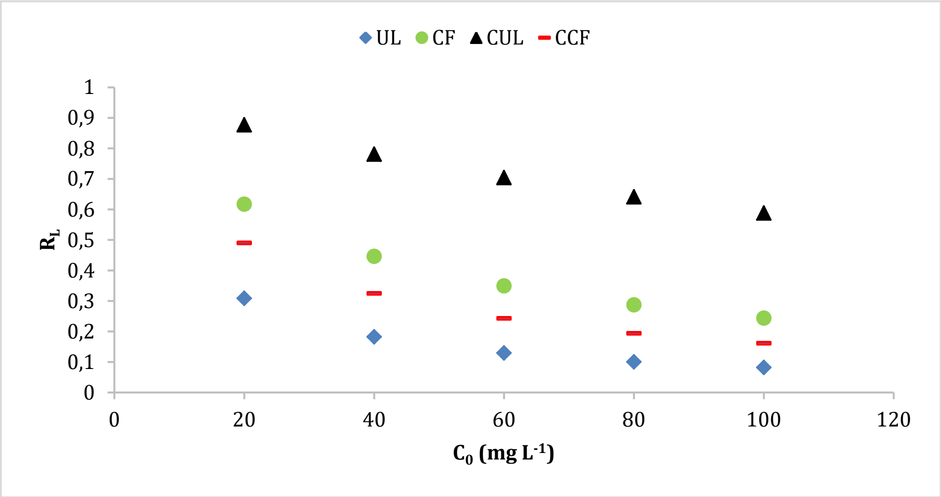

Fig 3. The equilibrium parameter (RL) of RBBR adsorption onto adsorbents

Table 4. Adsorption capacity of various adsorbents for the adsorption of RBBR

Adsorbent	Adsorption capacity, mg g^{-1} or %	pH	Ref.
Immobilized *Scenedesmus quadricauda*	48.3 mg g^{-1}	2	[30]
Jatropha curcas pods based activated carbon	95%	3	[31]
Bone char	20.6 mg g^{-1}	7.1	[32]
Sewage sludge based active carbon	33.47 mg g^{-1}	2	[33]
Peach-Palm (*Bactris gasipaes*) residue	1.8 mg g^{-1}	6.2	[34]
Poly(NOPMA)	238.10 mg g^{-1}	7	[35]
Carboxylated multi-walled carbon nanotubes	109.41 mg g^{-1}	4	[36]
P. eryngii immobilized on Amberlite XAD-4	98%	5.5	[37]
Pineapple leaf powder	96.2 %	-	[38]
Lime peel powder	95.9 %	-	[38]
Orange peel	95.72 %	-	[39]
Spent tea leaves	99.02 %	-	[39]
Carbonized sewage sludge	34.60 mg g^{-1}	3	[2]
Ulva lactuca	0.953 mg g^{-1}	3	This study
Codium fragile	0.683 mg g^{-1}	3	This study
Carbonized *Ulva lactuca*	2.113 mg g^{-1}	3	This study
Carbonized *Codium fragile*	1.997 mg g^{-1}	3	This study

4. CONCLUSIONS

RBBR adsorption from an aqueous solution by UL, CF, CUL and CCF was investigated in this study. According the obtained results, CUL showed the best adsorption performance (2.113 mg g^{-1}) while CF showed worst performance (0.683 mg g^{-1}) among the four samples. Besides, it was found that the equilibrium time was determined as 90 min for raw macroalgae and 60 min in the case of carbonized macroalgae. Adsorption kinetics were described better by the pseudo-second-order model rather than pseudo-first-order model. The best fit of equilibrium data was described by the Langmuir model for all adsorbents.

As can be seen from the results, adsorption capacities of the raw and carbonized macroalgae are not so good in comparison with the literature studies. However, adsorption capacities of these materials can be improved by activation of these materials directly, or after carbonization process. Therefore, it can be suggested that, further studies should be carried out to utilize these materials which are considered as waste, and produce new adsorbents.

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