Characterization of Carbons Produced from Gelidium Corneum and Adsorption of Crystal Violet

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Due to high production costs, the production of activated carbon from waste has attracted a lot of attention recently. In this study, gelidium corneum (GC) was carbonized at 800 °C for 90 min. Its carbonization yield, adsorption capacity, and physical and chemical properties were investigated. Ultraviolet-visible spectroscopy (UV), X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), energy-dispersive X-ray spectroscopy (EDX), scanning electron microscopy (SEM), proximate analysis and ultimate analysis were performed. According to XRD analysis, the structure of GC is semi-crystalline, but the crystalline structure increases after carbonization. The carbonization yield of GC was about 39%. According to SEM, UV and XRD analysis, the carbonization process supported crystallinity and the formation of micropore/mesopore structures. The crystal violet (CV) removal and adsorption capacity were 96 % and 9.63 mg/g at an initial dye concentration of 50 mg/L, 30 °C, adsorbent dosage of 5 g/L, constant stirring speed of 200 rpm and equilibration time of 60 min, respectively. The carbonized gelidium corneum (cGC) can be used as a suitable adsorbent for the removal of dyes from aqueous solutions. It can also be an alternative product to commercial products because of its high adsorption capacity and cost-effectiveness.

Keywords: gelidium corneum, activated carbon, adsorption, crystal violet.

1. INTRODUCTION

Industrial wastes contain significant concentrations of dye [1]. Many methods, including chemical reduction, electrochemical purification, evaporation, chemical precipitation and adsorption, reverse osmosis, filtration, and ion exchange, are used to remove water contaminants [2 – 4]. Most of these are generally limited in their use due to their high cost, energy consumption, and the generation of large amounts of toxic waste [5]. However, activated carbons produced from bio-based wastes eliminate some of these disadvantages. Activated carbons, produced from many sources such as biomass and waste plastics, have a high surface area [6, 7]. Therefore, they are widely used in many applications such as separation/purification of gases and liquids, production of composite materials, catalyst/catalyst support, removal of toxic substances, supercapacitors, and electrodes [8 – 13]. The physical/chemical activation processes are generally applied to increase the surface area and surface activity of activated carbons [14]. After the carbonization process, numerous micropores, mesopores, and macropores are formed within their structures [15, 16]. If the physical/chemical activation is not done before/after carbonization, micropores may not form significantly [17, 18]. While the adsorption capacity of the carbons obtained from waste onions is 8.7 mg/g, the adsorption capacity of their activation with KOH is 18.6 mg/g [19]. Dil et al. [20] found that the CV dye adsorption of a commercial activated carbon was 35 mg/g, while zinc (II) oxide nanorod loaded on activated carbon (ZnO-NRs-AC) was 81 mg/g. Guo et al. suggested that sulfuric acid (H_2SO_4)-activated carbons are more effective than CO_2-activated carbons for removing ammonia (NH_3). However, this improvement is not solely dependent on their surface area [21]. On the other hand, these processes could seriously increase production costs [22]. To produce an economical adsorbent, cost-increasing additional processes should be avoided as much as possible. Therefore, in this study, waste biomass (gelidium corneum) was carbonized directly (only dried in the sun) without washing or drying, and no chemical or physical activation process was applied. The effect of the carbons on the removal of crystal violet (CV) from wastewater was then determined. Gelidium corneum (GC) is a well-known red seaweed that grows on sea coasts [23]. Many commercial products, such as agar, are derived from gelidium and are utilized in various industries, particularly in cosmetics and food [24, 25]. However, there are no available studies on the removal of CV from carbonized gelidium corneum (cGC). This study examined the physical and chemical properties of cGC and its potential for removing CV dye.

CV is a cationic dye widely used in the industry [26]. Cationic dyes may easily interact with cell membrane surfaces and may also concentrate in the cytoplasm inside the cell, making them more harmful than anionic dyes [27, 28]. Additionally, CV is widely used in applications such as medicinal solutions and animal feeds [29]. However, these applications result in the generation of dye effluent, which is discharged into the sea and lakes through the sewage system. CV is considered harmful to biological life as it is a strong carcinogen and promotes tumor growth [30, 31]. Therefore, the removal of CV from wastewater is crucial for the environment and biological life.

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2. MATERIAL AND METHOD

2.1. Material

In experimental studies, CV (Carlo Erba, purity: 99 %, C.I.:42555, Cas No: 548-62-9), whose molecular weight is 407.98 g/mol and empirical formula is $C_{25}H_{30}N_3Cl$ [32], was used as adsorbate. CV solution was prepared by dissolving it in 1000 mg/L of distilled water. GC gathered on the shores of the Marmara Sea in Istanbul, Turkey. GC contains moisture of 6.0 %, ash of 22.4 %, volatile matter of 68.3 %, and fixed carbon of 3.3 % (Table 1). As seen in the ultimate analysis of GC in Table 1, it contains carbon (C) of 26.6 %, hydrogen (H) of 4.5 %, oxygen (O) of 62.1 %, nitrogen (N) of 2.6 %, and sulphur (S) of 4.2 %. The carbon content in the material is low but still higher than most biomass [33]. In addition, Fig. 1 shows the results of the EDX analyses performed on the 10 µm surfaces of the samples.

The GC samples also contain calcium (Ca), potassium (K), silicon (Si), aluminum (Al), magnesium (Mg), phosphorus (P), chlorine (Cl), and sodium (Na).

Table 1. Ultimate and proximate analysis

Proximate analysis, wt.%		Ultimate analysis, wt.%, daf		Carbonization yield, %
Moisture	6.0	Carbon (C)	26.6	38.60
Ash	22.4	Hydrogen (H)	4.5	
Volatile matter	68.3	Nitrogen (N)	2.6	
Fixed carbon*	3.3	Sulfur (S)	4.2	
		Oxygen (O*)	62.1	
*Calculated by difference				

2.2. Methods

2.2.1. Analysis

The microstructure of the natural gelidium corneum (GC) and the carbonized gelidium corneum (cGC) were examined by scanning electron microscopy (SEM, LEO EVO 40). The quantitative determinations of GC and the cGC were made with PerkinElmer Spectrum One FTIR (650 – 4000 cm^{-1}, 2 cm^{-1} resolution and 128 scanning). Adsorption experiments were implemented with a UV-visible spectrophotometer (Shimadzu UV-1700 Pharmaspec) at 590 nm.

2.2.2. Experiments

The experimental procedure is given in Fig. 2.

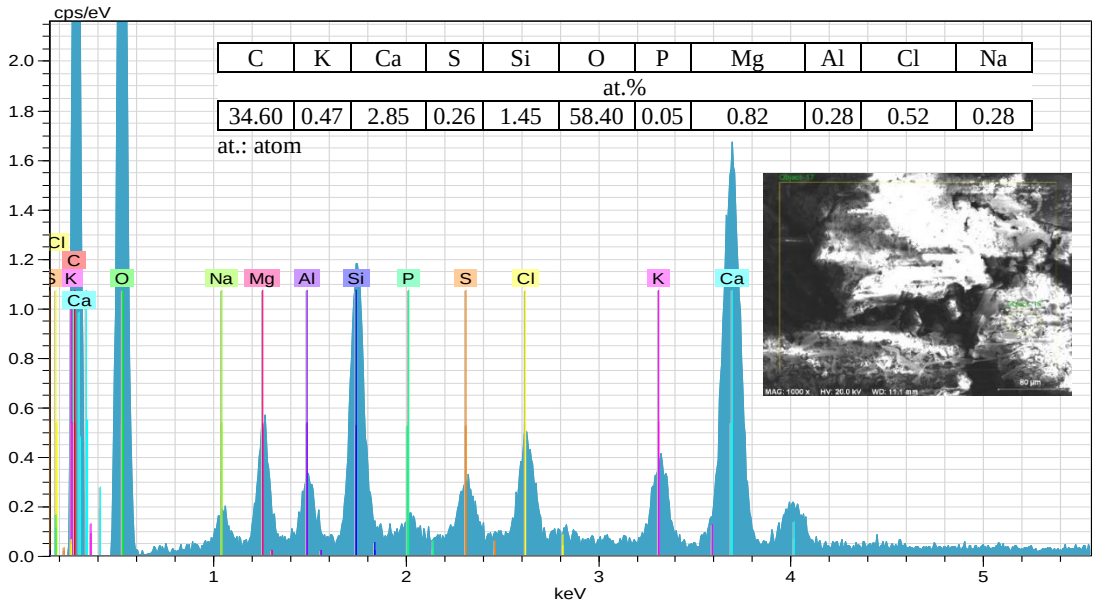


Fig. 1. EDX spectrum of GC

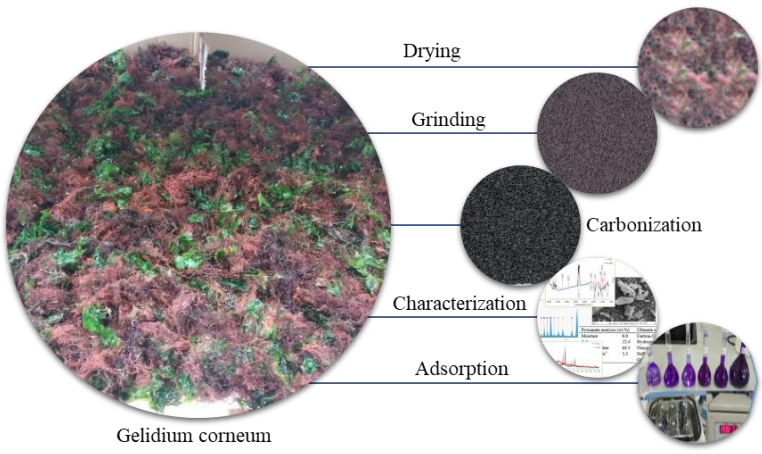


Fig. 2. Experimental procedure

The equilibrium adsorption, qe (mg/g), was calculated using Eq. 1, and the percentage of CV removal (%) was determined using Eq. 2.

$$q_e = \frac{Ci-Ce}{m}.V; \quad (1)$$

$$Removal\ (\%) = \frac{Ci-Ce}{Ci}.100, \quad (2)$$

where, *qe* (mg/g) represents the amount of dye adsorbed per unit mass of the cGC. *Ci* (mg/L) is the initial dye concentration of *CV*, and *Ce* (mg/L) is the equilibrium adsorption of *CV*, and *m* (g) stands for the amount of the cGC, and *V* (L) represents the volume of the solution.

The carbonization yield of GC (Gy) was calculated according to Eq. 3.

$$\text{Gy}\ (\%) = \frac{W_a}{W_b} \times 100, \quad (3)$$

where, *Wb* (g) is the dry weight of GC, and *Wa* (g) is the weight of carbonized GC.

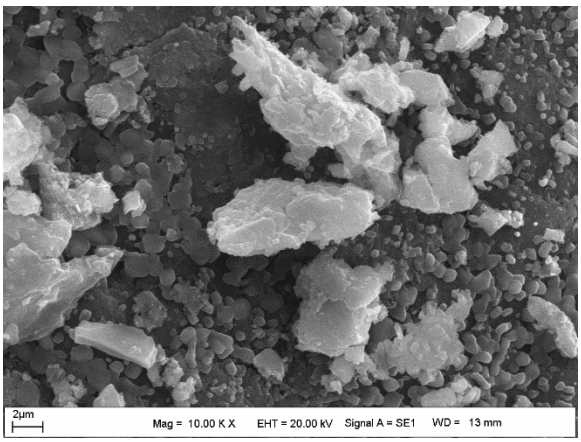
3. RESULTS AND DISCUSSION

3.1. Characterization of adsorbent

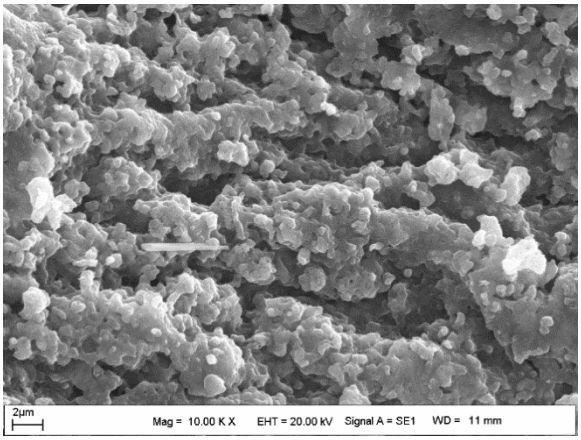
The carbonization yield of GC was 38.60 % under these conditions (Table 1). Duckweed's carbonization yield is 34 % [32]. Slash pine wood's carbonization yield is 28 % [34]. The carbonization yield of rice husk chemically activated with sulfuric acid (H_2SO_4) and zinc chloride ($ZnCl_2$) is 36 % and 32 %, respectively [35]. In summary, it can be said that the carbonization yield of GC is satisfactory.

The structure of GC and the cGC were analyzed by SEM (LEO-EVO 40). SEM analyses of GC before and after carbonization are given in Fig. 3. As seen in Fig. 3 a, there were no porous structures in the surface morphology of GC before carbonization, while porous structures were observed after carbonization (Fig. 3 b). SEM micrograph of the cGC showed the presence of pores of different sizes, indicating that very rough and heterogeneous textures were formed. This is highly beneficial as it provides greater surface availability for adsorption, thus increasing the adsorption efficiency of CV [36, 37]. Fourier transform infrared (FTIR), which is used as a qualitative technique [38], was applied to determine the functional groups on the surface of gelidium corneum (GC) before and after carbonization. As seen in the FTIR spectrum of GC given in Fig. 4, O-H stretching at 3265 cm^{-1} [39], C-H stretching at 2927 cm^{-1} [40, 41], C-H bending at 856 cm^{-1} [42], C=C stretching at 1635 cm^{-1} [43], C-C stretching at 1436 cm^{-1} [44], C-OH stretching at 1019 cm-1 [45], and C-O-C symmetrical stretching at 1229 cm-1 occurred [46]. Most of these bands disappeared after carbonization (cGC). This may have been caused by the high carbon content of the cGC.

As the ratio of elements such as hydrogen (H) and oxygen (O) in a substance decreases, their bands in the FTIR spectrum disappear [47]. After carbonization of the cGC, many of these peaks disappeared, and the hydroxyl group peak of 3286 cm^{-1} shifted to 3773 and 3656 cm^{-1}, and the C-O stretching peak of 1019 cm^{-1} shifted to 1107 cm^{-1}, and the C-O stretching peak of 1436 cm^{-1} shifted to 1430 cm^{-1}, and the C≡C stretching band of 2347 cm^{-1} appeared.



a



b

Fig. 3. SEM image of GC: a – before carbonization; b – after carbonization

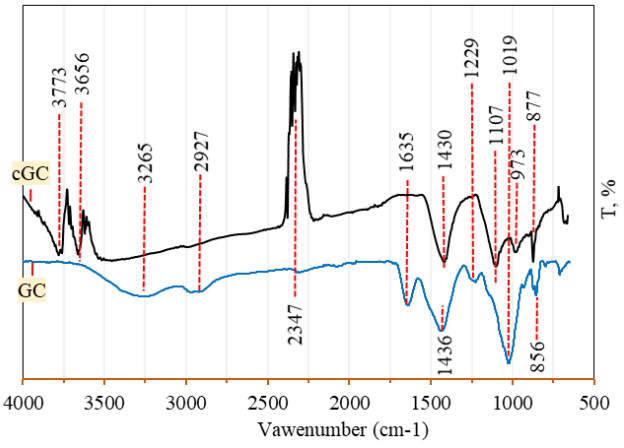


Fig. 4. FTIR analysis

The cGC appeared the strong bands at 2347 cm^{-1} (C≡C stretch) and 1107 cm^{-1} (C-O stretch), and weak bands at 3780 and 3652 cm^{-1} (O-H stretch). The cGC showed the presence of many functional groups, binding sites responsible for CV absorption.

The prediction of the adsorption mechanism of CV dye on the cGC surface is shown in Fig. 5. The positively charged groups of CV and the negatively charged groups of the cGC surface may contribute to the electrostatic forces [48].

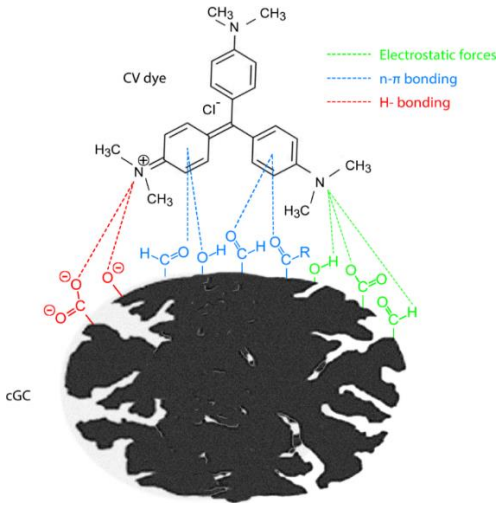


Fig. 5. Illustration of the possible interaction between the cGC surface and CV (H-bonding, n-π stacking, and electrostatic forces)

H-bonding may occur between the hydrogen on the surface of the cGC and the nitrogen atoms in the structure of the CV dye [49]. The n-π interaction may have played a role in CV adsorption through the interaction between the cGC surface and CV aromatic rings [50].

The physical properties, chemical composition and crystallographic structure of GC and the cGC were investigated by X-ray diffraction (XRD) analysis (Fig. 6). As can be seen in Fig. 6, in the XRD analysis of GC, a broad diffraction peak occurred around 2θ = ~22°, corresponding to the diffraction of the (002) planes, which corresponds to the typical graphite plane [51].

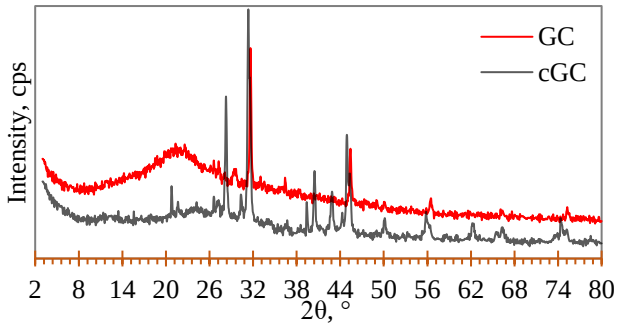


Fig. 6. XRD patterns of GC and cGC

The broadening of the peak indicates the presence of amorphous carbon and a low degree of graphitization [52]. In addition, distinctive diffraction peaks occurred at 31°, 45°, 56° and 75°. This may have been caused by the presence of many elements (C, O, H, K, Ca, S, Si, P, Mg, Al, Cl, Na) in the content of GC (Fig. 1). It can be said that GC has a semi-crystalline structure due to the formation of a wide diffraction peak at about 22°, and prominent peaks at 31°, 45°, 56° and 75°. However, carbonization of GC (cGC) at 800 °C, 2θ = 22° wide diffraction peaks almost disappeared, and 20°, 26°, 28°, 30°, 31°, 39°, 40°, 42°, 44°, 45°, 50°, 55°, 62° and 74° diffraction peaks occurred. In XRD phase analysis, it was indexed to correspond to sylvite (KCl) at 28° and 40° [53], and calcium sulfide (CaS) at 31°, 44°, 55° and 74° [54], and silicon tetrachloride (SiCl4) at 45° and 50° [55], and silica (SiO_2) at 20°, 26°, 30°, 39°, 42° [56, 57], and magnesium oxide (MgO) at 62° [58]. The presence of these elements was supported by EDX analysis (Fig. 1). This shows that the graphite structure is destroyed and porous structures are formed [59], which is quite compatible with the SEM image given in Fig. 3.

3.2. Adsorption capacity of adsorbent

CV dye removal of carbonized GC (cGC) and the results obtained in current studies are given in Table 2. Adsorption experiments were carried out under specified conditions, which were 30 °C, pH:6, adsorbent dosage of 5 g/L, contact time of 60 min, and initial concentration of 50 mg/L. The removal (%) and adsorption capacity were 96 % and 9.63 mg/L, respectively. Activated with NaOH, the NAJL achieved 98 % removal with the adsorbent dosage of 0.4 g/L, while the cGC was 96 % at the adsorbent dosage of 5 g/L (Table 2). Chemical and physical activation can improve the pore structure and surface areas of carbons, although they increase the cost of adsorbent production [60, 61]. The cGC provided more CV removal than the cWC. The activated carbon lemon wood/Fe_3O_4 provides 98 % removal at the initial concentration of 10 mg/L, while the cGC can reach at higher (50 mg/L) initial concentrations. Compared to the existing adsorbents used in CV removal given in Table 2, it can be said that the adsorption capacity of the cGC is satisfactory.

Table 2. Removal of CV

Sample name	Chemical/physical treatment	Adsorbent dosage, g/L	Initial concentration, mg/L	Removal, %	*qe*, mg/g	
cWC (Carbonized waste coffee)	none	5	50	25	2.52	[62]
cDW (Carbonized duckweed)	none	5	50	96	9.62	[32]
Activated carbon lemon wood/Fe_3O_4	commercial	5	10	99	~2	[63]
Functionalized multi-walled carbon nanotubes (fMWNTs)	commercial	0.5	50	82	90	[64]
NaOH-activated aerva javanica leaf (NAJL)	NaOH	0.4	50	98	3.7	[65]
PLAC (Poultry litter activated carbon)	$ZnCl_2$	2.5	50	92	70.3	[66]
cLM/HS (Carbonized hazelnut shell and lemna minor)	none	1	100	88	87.95	[67]
Fe_3O_4-MNPs (Magnetic nanoparticles modified with $NaC_{12}H_{25}SO_4$)	NaOH	0.25	10	80	166.67	[68]
cGC	none	5	50	96	9.63	This study

The adsorption capacity of the cGC can be further increased by determining adsorption parameters such as absorbent dose, initial dye concentration, contact time, pH, and temperature.

4. CONCLUSIONS AND SUGGESTIONS

In this study, gelidium corneum (GC) carbonized at 800 °C for 90 min was characterized and its effectiveness was determined by the removal of crystal violet (CV) found in industrial wastewater. The obtained results are provided below.

1. Proximate analysis shows that the GC contains 22.4 % ash, 6.0 % moisture, 3.3 % fixed carbon, and 68.3 % volatile matter.
2. Ultimate analysis indicates that GC consists of 26.6 % C, 4.5 % H, 2.6 % N, 4.2 % S, and 62.1 % O.
3. According to EDX analysis, GC contains various elements such as C, K, Ca, S, Si, O, P, Mg, Al, Cl, and Na.
4. The carbonization yield of GC is 38.60 %.
5. FTIR analysis shows that the peaks of the functional groups of GC changed after carbonization.
6. SEM analysis reveals the formation of numerous porous structures after carbonization.
7. According to XRD analysis, the semi-crystalline structure of GC changed significantly to the crystalline structure after carbonization.
8. The cGC absorbent demonstrated a 96 % removal rate and an adsorption capacity of 9.63 mg/L for CV dye.

The cGC absorbent was proven to be both effective and inexpensive in the removal of CV dye from wastewater. Further research is needed to determine the optimal adsorption parameters for the cGC absorbent in CV removal.

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