

Activated carbon from gypsophila roots: a sustainable adsorbent for dye removal

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This study investigates the production and characterization of activated carbon derived from the roots of *Gypsophila* spp., an endemic genus of flowering plants belonging to the Caryophyllaceae family. Due to their high saponin content and abundant biomass in the Anatolian region of Türkiye, *Gypsophila* roots represent a sustainable precursor for carbon-based adsorbent materials. In this work, the effects of activating agent type and dosage on pore formation during the preparation of activated carbon from *Gypsophila* roots were examined. For this purpose, chemical activation was carried out using potassium hydroxide (KOH), phosphoric acid (H₃PO₄), sulfuric acid (H₂SO₄), and citric acid (C₆H₈O₇) at various concentrations. To achieve the desired porous structure, the process parameters activation temperature, activation time, and impregnation ratio were optimized using the Box–Behnken experimental design. The activated carbons produced under optimal conditions were characterized using Brunauer–Emmett–Teller (BET) surface area analysis and a series of complementary tests to evaluate their textural properties. The sample exhibiting the most developed porous structure was selected for further adsorption studies. In particular, the performance of the *Gypsophila*-derived activated carbon in removing cationic and anionic dyes from aqueous solutions will be investigated to assess its potential application in wastewater treatment.

Keywords: *Gypsophila* spp., activated carbon, wastewater treatment, dye, environmental

INTRODUCTION

Dye compounds are essential chemicals widely used in various industries such as textiles, paper, plastics, and cosmetics, and they represent one of the major pollutants in industrial wastewater [1]. Due to their high solubility in water, chemical stability, and resistance to biological degradation, these compounds are considered particularly hazardous organic pollutants to the environment [2,3]. Therefore, the removal of dyes from wastewater is of great importance in terms of environmental protection and sustainable water management. Various chemical and physical treatment methods such as flocculation, coagulation, sedimentation, ozonation, and adsorption are commonly employed for this purpose [2,4]. However, adsorption using activated carbon has been shown to be a more effective and economical treatment technique compared to other methods, owing to its high efficiency in removing a wide range of adsorbates, operational simplicity, and ease of design [1,4,5].

Activated carbon (AC) is a carbon-based material characterized by its high porosity, large surface area, high adsorption capacity, mechanical strength, and surface reactivity [6]. Nevertheless, the high cost of commercial activated carbon and its reliance on non-renewable precursors have prompted researchers to

explore low-cost, sustainable alternatives derived from agricultural and natural biomass [4,7,8]. In recent years, growing research interest has been directed toward the production of activated carbon from renewable biomaterials such as coconut husk [4], palm seed shells [9], jute fibers [10], olive stones [11], plum kernels [12], and bamboo [13].

The *Gypsophila* plant (*Gypsophila* spp.), belonging to the Caryophyllaceae family, is a perennial herbaceous species that grows 40–100 cm tall, has a deep taproot system, and produces white flowers between June and July [14,15]. Native to the Mediterranean basin, Western Asia, and parts of Europe, *Gypsophila* is widely distributed in Türkiye, where it is also considered an economically valuable export product. The high saponin content found particularly in its roots is the main factor contributing to its commercial importance [16–18]. Owing to its high saponin concentration and abundant root biomass, *Gypsophila* species can be regarded as promising and sustainable raw materials for various industrial and environmental applications. However, despite their availability and chemical richness, studies investigating the use of *Gypsophila* roots as precursors for activated carbon production remain limited in the literature. This makes the utilization of this plant both innovative and environmentally significant.

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In this study, *Gypsophila* roots were selected as a novel and sustainable precursor for the production of activated carbon. Since the adsorption capacity of activated carbon is highly influenced by preparation conditions, optimization of the production process is essential [6]. For this purpose, chemical activation was carried out using potassium hydroxide (KOH), phosphoric acid (H₃PO₄), sulfuric acid (H₂SO₄), and citric acid (C₆H₈O₇) as activating agents. To optimize the structural and adsorption properties of the obtained materials, key process parameters activation temperature, activation time, and impregnation ratio were systematically investigated using response surface methodology (RSM). RSM is a statistical optimization technique used to evaluate the effects of independent variables on response outputs, and the Box–Behnken design (BBD) is an efficient approach within this methodology that enables the determination of optimum conditions with fewer experimental runs [19]. After determining the optimal conditions, the resulting activated carbons were characterized using various analytical techniques.

This study aims to address the existing gap in the literature regarding the utilization of endemic Turkish plant species as precursors for activated carbon production and to contribute to the development of cost-effective, sustainable, and high-performance adsorbents for the removal of both cationic and anionic dyes from textile wastewater.

MATERIALS AND METHODS

Gypsophila roots and cultivation conditions

Roots of *Gypsophila* were procured in Kalealtı Çöğen and Dried Nuts Import Export Industry and Trade Ltd. Co. (Kalealtı Çöğen ve Kuruyemiş İth. İhr. San. ve Tic. Ltd. Şti.). The roots of *Gypsophila* were subjected to a grinding process in an industrial disc mill operating at a high speed. This resulted in the formation of particles measuring between 3-5 millimeters in size, which were then prepared for experimental use.

Preparation of activated carbon

The following substances were utilised as activation agents for the activation of *Gypsophila* root: potassium hydroxide (KOH), sulfuric acid (H₂SO₄), citric acid (C₆H₈O₇), and phosphoric acid (H₃PO₄). The root mass of *Gypsophila* was impregnated with activation agents at ratios of 0:1, 0:3, and 0:5 (w:w). The impregnated *Gypsophila* root was subjected to a 24-h mixing process in a shaker. Subsequently, the samples were subjected to filtration and maintained within an oven (NUVE FN 500 dry air

sterilizer) set at 60°C until a complete desiccation had been achieved. The activated carbons were finally ground.

Impregnated samples obtained from *Gypsophila* roots were evaluated in terms of various process parameters to optimize the preparation conditions of activated carbon after the activation process (Table 1). In this study, the primary factors presumed to influence adsorption capacity were identified as impregnation ratio, carbonization temperature, and carbonization time. Consequently, root samples that had been treated with a range of activation agents and impregnation ratios were subjected to carbonization at varying temperatures and times. The performance of the resulting activated carbons was then evaluated based on their adsorption capacities.

Table 1. Factors of carbonization of *Gypsophila* root

Levels	Impregnation ratio (w:w)	Carbonization time (min)	Carbonization temperature (°C)
1	0:1	30	450
2	0:3	60	525
3	0:5	90	600

Adsorption studies

- *Methylene blue test.* Methylene blue (MB) (methylthionium chloride) was utilised as an adsorbent. Samples of the optimised AC, with a mass of 10 mg, were introduced to 25 mL of dye solution, which had an initial concentration of 100 mg/L. The suspension was stirred for 24 h in order to achieve equilibrium. Subsequently, the solution was separated from the sorbent through centrifugation at 5000 rpm for 5 min. The concentration of the dye solutions was determined by means of a UV-Vis spectrophotometer. Adsorption capacity (q) was calculated using Equation 1.

$$q = \frac{C_0 - C_e \cdot V}{m} \quad (1)$$

where q is adsorption capacity (mg/g), C₀ is initial methylene blue concentration (mg/L), C_e is the methylene blue concentration after adsorption process has been completed (mg/L), V is solution volume (L), m is activated carbon amount (g).

Based on the methylene blue results, the samples exhibiting the best performance for each chemical were selected and characterized using iodine number analysis.

- *Iodine number test.* Iodine number analysis was carried out to determine the adsorption capacity of the activated carbon samples. In order to achieve this objective, 0.1 N sodium thiosulphate, 0.1 N

iodine solution and a 1% starch indicator were meticulously prepared. For the 0.1 N sodium thiosulphate solution, 6.21 g of $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ was dissolved in distilled water, and the volume was made up to 250 mL. The 0.1 N iodine solution was prepared by means of the following procedure. A quantity of 5–6 g of KI was dissolved in 10–15 mL of distilled water, after which 3.17 g of I_2 was added. The solution was stirred using a magnetic stirrer until no iodine crystals remained, at which point the volume was made up to 250 mL. The solution was then stored in an amber bottle. For the experiment, 0.1 g of each activated carbon sample was weighed into falcon tubes, and 25 mL of 0.1 N iodine solution was added to each tube. A control sample without activated carbon was also prepared under the same conditions. The tubes were mixed in an orbital shaker at 170 rpm for 2 hours. At the end of the period, the samples were centrifuged at 5000 rpm for 5 minutes to allow the solid phase to settle.

Following centrifugation, 10 mL of each sample's superior layer was extracted and titrated with a 0.1 N sodium thiosulphate solution. During the titration process, when the solution assumed a pale straw-yellow hue, 3–4 drops of 1% starch indicator were introduced, and the titration was continued until the dark blue colour was fully dissipated. The volumes of sodium thiosulphate consumed were recorded, and the iodine adsorption capacity of the activated carbons was calculated by comparison with a blank sample.

Statistical analysis

In order to systematically investigate the effects of three parameters (impregnation ratio, carbonization temperature, and carbonization time) which have been identified as the most important factors affecting adsorption capacity in activated carbon production from *gypsophila* roots, and to optimise the number of experiments, a Box-Behnken experimental design (BBD) was used, with adsorption capacity (q) evaluated as the response variable. A second-order mathematical model was developed to predict adsorption capacity using the experimental data obtained with BBD. However, an examination of the model outputs revealed that the Box-Behnken approach did not adequately represent the adsorption system. This phenomenon is hypothesized to be attributable to the non-dependence of the adsorption mechanism on a single experimental parameter. Rather, it is theorized that multiple factors, including pore structure, surface functional groups, carbonization degree, and adsorbent-adsorbate interactions, collectively exert an effect. Consequently, it was determined that the

classical RSM approach is constrained by the nonlinear and complex nature of the system. To address this limitation, machine learning methods were employed in the subsequent stages of the study.

RESULTS AND DISCUSSION

Methylene blue adsorption results

The adsorption performance of activated carbons produced from *Gypsophila* roots using different chemical activation agents was evaluated through methylene blue (MB) adsorption experiments. The resulting adsorption capacity data are presented in Fig. 1.

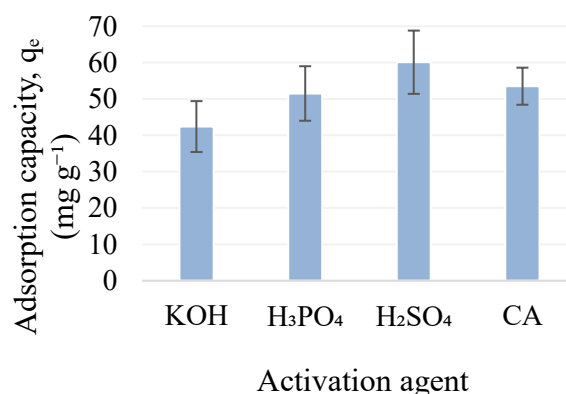


Figure 1. Effect of activation agent on methylene blue adsorption capacity.

The average adsorption capacity of activated carbons treated with H_2SO_4 was approximately 60 mg g^{-1} . The lowest adsorption performance was observed for KOH activated samples, with an average adsorption capacity of approximately 42 mg g^{-1} . Samples activated with H_3PO_4 and citric acid exhibited moderate adsorption performance.

The higher adsorption performance of H_2SO_4 activation can be attributed to sulfuric acid due to the strong dehydration effect during carbonization, resulting in a more developed pore structure. Oxygen-containing surface functional groups formed during H_2SO_4 activation may enhance electrostatic interactions with cationic methylene blue molecules.

In contrast, excessive pore expansion or partial collapse of the carbon structure may occur during KOH activation at high temperatures, resulting in lower adsorption capacity.

The highest adsorption capacity obtained in the study was 78.5 mg g^{-1} , achieved in H_2SO_4 activated carbon prepared at 525°C , with a 30-min activation time, and a 0.1 impregnation ratio. This result suggests that low impregnation rates may be suitable for effective pore formation.

Evaluation of process parameters using the Box–Behnken experimental design

A Box-Behnken experimental design was applied to investigate the effect of activated carbon production parameters on adsorption performance. The three parameters considered to have the greatest impact on adsorption performance were identified as temperature, activation time, and impregnation ratio. Accordingly, a range of 450–600 °C for temperature, 30–90 min for time, and 0.1–0.5 for the impregnation ratio was selected (Table 2).

Table 2. Box-Behnken experimental design plan for activated carbon production

Run	Temperature (°C)	Time (min)	Impregnation ratio
1	450	30	0.3
2	600	30	0.3
3	450	90	0.3
4	600	90	0.3
5	450	60	0.1
6	600	60	0.1
7	450	60	0.5
8	600	60	0.5
9	525	30	0.1
10	525	90	0.1
11	525	30	0.5
12	525	90	0.5
13	525	60	0.3
14	525	60	0.3
15	525	60	0.3

The experimental results show that adsorption capacity is significantly influenced by the process parameters. In general, increasing the activation temperature improved adsorption performance by promoting pore formation. However, excessively high temperatures had the opposite effect, causing pore collapse in some samples and consequently reducing adsorption capacity.

Similarly, increasing the activation time did not always lead to a linear increase in adsorption capacity. In some cases, longer carbonization times resulted in a reduction of the active surface area, thereby limiting adsorption performance.

The results also indicated that the impregnation ratio played an important role in determining adsorption performance. While more balanced pore development is achieved at low impregnation ratios, excessive chemical degradation of the carbon structure may occur at high impregnation ratios.

The RSM model did not adequately represent the adsorption system. The nonlinear behavior of the system may be associated with the combined effects of pore structure, surface functional groups, carbonization degree, and adsorbent–adsorbate interactions. These results suggest that classical RSM approaches may be insufficient for modeling complex adsorption systems involving multiple interactions. Therefore, machine learning approaches were adopted to better model the adsorption system.

Machine learning modeling of adsorption performance

Due to the limitations of the classical RSM approach, machine learning methods were evaluated to model adsorption performance. In this context, linear model (LM), support vector machine (SVM), GLMNET, and random forest (RF) algorithms were compared.

The performance results for the evaluated models are shown in Figure 2. Among the evaluated models, RF demonstrated the highest predictive performance, with an R^2 of 0.7295 and an RMSE of 5.3159.

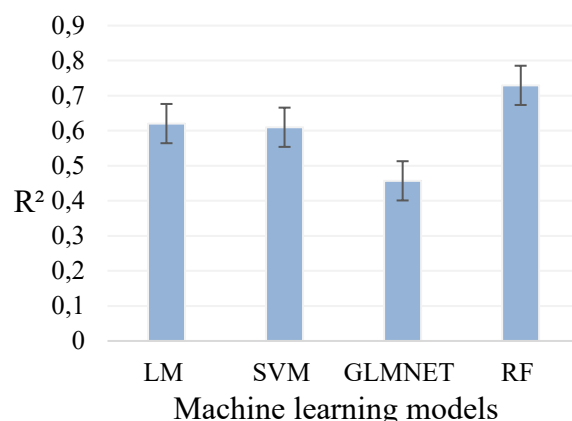


Figure 2. Comparison of machine learning model performances based on R^2 values.

Unlike traditional statistical models, the Random Forest model has yielded more successful results because it can more effectively model the complex, nonlinear interactions between process parameters and adsorption capacity. The optimum production conditions were determined as follows:

- H_2SO_4 activation,
- 0.1 impregnation ratio,
- 525–600 °C temperature range,
- 60–90 min activation time.

These results indicate that machine learning approaches can provide more reliable predictions for complex adsorption systems.

Iodine number analysis of selected activated carbons

The activated carbon samples exhibiting the highest methylene blue adsorption performance were examined in greater detail using iodine number analysis to evaluate the development of microporosity. The resulting iodine number values are presented in Figure 3.

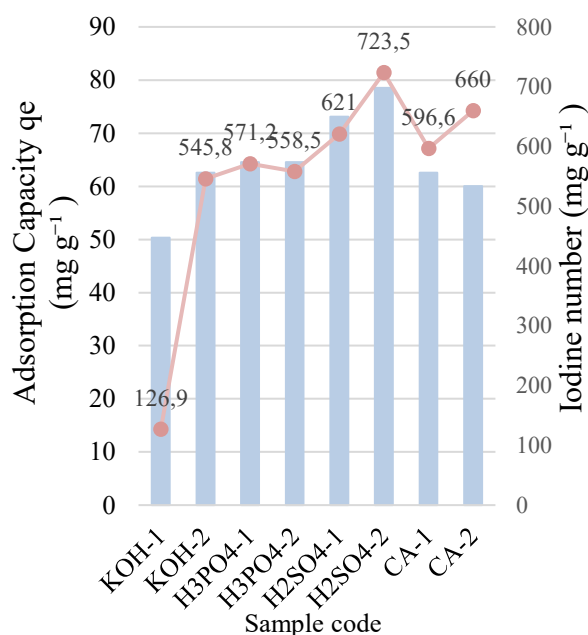


Figure 3. Comparison of adsorption capacity and iodine number values of selected activated carbon samples.

The iodine number values of the samples range from 126.9 to 723.5 mg g^{-1} . The highest iodine number was obtained from the sample activated with H₂SO₄, and this result indicates that activation with H₂SO₄ promotes the formation of micropores.

Owing to their small size, iodine molecules are primarily adsorbed in micropores. Therefore, high iodine numbers are generally associated with high micropore volume and increased surface area.

The results generally indicate a parallel relationship between the iodine number and the methylene blue adsorption capacity. However, it was found that this relationship is not entirely linear. Since methylene blue molecules are larger in size compared to iodine molecules, it is believed that the mere presence of micropores is insufficient. It is believed that mesopores and an appropriate pore size distribution are also important for the effective adsorption of methylene blue. The fact that the

adsorption capacity remains limited in some samples despite a high iodine number may be due to the pore entrances not being sufficiently accessible to large dye molecules. These results demonstrate that the coexistence of micro- and mesopores is important in dye adsorption applications.

BET surface area analysis

The textural properties of the selected activated carbon samples were determined using nitrogen adsorption–desorption analyses. The micropore and BET surface area results for the samples are presented in Table 3. It is observed that the BET surface areas of the samples range from 5.17 to 87.90 m^2/g . The highest BET surface area of 87.90 m^2/g was obtained for the H₂SO₄-1 sample, followed by the H₃PO₄-2 (65.71 m^2/g) and CA-2 (54.98 m^2/g) samples. The lowest surface areas were determined for the KOH-1 and KOH-2 samples.

Table 3. Textural properties of activated carbon samples determined by BET analysis

Sample code	BET surface area (m^2/g)	Micropore area (m^2/g)
KOH-1	5.17	–
KOH-2	5.66	1.04
H ₃ PO ₄ -1	10.72	0.84
H ₃ PO ₄ -2	65.71	41.69
H ₂ SO ₄ -1	87.90	59.81
H ₂ SO ₄ -2	22.60	6.49
CA-1	40.33	15.40
CA-2	54.98	36.94

When the BET results were evaluated in conjunction with iodine number analyses, a direct relationship was not observed between the two methods. Although the highest BET surface area and micropore area were obtained for the H₂SO₄-1 sample, the highest iodine number was determined for the H₂SO₄-2 sample. This suggests that iodine adsorption is influenced not only by the total surface area but also by the pore size distribution and surface properties. However, it can be stated that samples with high BET surface areas generally exhibit more developed pore structures and support adsorption performance.

In conclusion, the H₂SO₄-1 sample exhibited the most advanced textural properties among the studied samples due to its highest BET surface area and micropore area.

Comparison with literature

The adsorption capacity and iodine number results obtained in this study were compared with

those reported in the literature for biomass-based activated carbons (Table 4).

The methylene blue adsorption capacity of activated carbons derived from *Gypsophila* roots ranged from 50 to 78 mg g⁻¹, showing similar performance to sawdust-based activated carbons. However, lower capacity values were obtained compared to highly porous activated carbons derived from certain biomass sources such as coconut shells, rice husks, and corn cobs.

Table 4. Comparison of biomass-derived activated carbons reported in the literature

Precursor	Activation	MB adsorption (mg g ⁻¹)	Iodine number (mg g ⁻¹)	Ref.
Various biomass	Chemical / thermal	30–50	500–1200	[7]
Sawdust	ZnCl ₂	78	650	[20]
<i>Gypsophila</i> (This study)	Chemical activation	50–78	546–723	<i>This work</i>
Corn cob	KOH	92	850	[21]
Rice husk	H ₃ PO ₄	104	720	[22]
Coconut shell	Steam activation	120–150	900–1100	[23]

The iodine number values, fell within the typical range reported in the literature for biomass-based activated carbons. In particular, the fact that the highest iodine number was at a level similar to that of rice husk-based activated carbon indicates successful microporous development.

The results obtained demonstrate that *Gypsophila* roots are a suitable, low-cost, and sustainable biomass source for activated carbon production.

CONCLUSION

In this study, activated carbon was successfully produced from *Gypsophila* roots using a chemical activation method. The adsorption performance of the produced activated carbons was evaluated using methylene blue.

It was determined that the activation agent had a significant effect on adsorption performance. Among the chemicals examined, the highest adsorption capacity was obtained in samples activated with H₂SO₄. The highest adsorption capacity was determined to be 78.5 mg g⁻¹, and this value was obtained from a sample prepared at 525 °C, with an activation time of 30 minutes and an impregnation ratio of 0.1.

The effects of process parameters were evaluated using a Box–Behnken experimental design.

However, it was determined that the classical RSM approach could not adequately represent the adsorption behavior due to the system's nonlinear nature.

For this reason, various machine learning models were applied, and the best predictive performance was achieved using the Random Forest model. For the RF model, the R² value was calculated as 0.7295 and the RMSE value as 5.3159.

It was observed that the iodine number analysis applied to the selected samples yielded results similar to those obtained from methylene blue adsorption following H₂SO₄ activation. BET analysis revealed that H₂SO₄-1 possessed the highest BET surface area (87.90 m² g⁻¹) and micropore area (59.81 m² g⁻¹), indicating the development of a more porous structure compared with the other activated carbons.

The results indicate that *Gypsophila* roots are a low-cost and sustainable biomass source for activated carbon production. Furthermore, it was determined that machine learning methods constitute an effective approach for modeling complex adsorption systems.

Future studies are recommended to focus on enhancing mesopore formation, investigating adsorption performance with different pollutants, and testing in real-world wastewater applications.

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