



Effect of different activation agents on the pollution removal efficiency of date seed activated carbon: process optimization using response surface methodology

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Abstract

Activated carbons are currently one of the most widely used materials for water and wastewater treatment because of their high specific surface area and moderate cost. This study is about the comparison of different activation methods on the pollution removal efficiency of date seed activated carbon using response surface methodology (RSM). The date seeds were carbonized in a muffle furnace at 300 °C for 1 h to produce carbon. Then, the chemical activation was carried out using 1 N solution of H₃PO₄, HNO₃, H₂SO₄ separately for 24 h. Activated carbons were ground in a grinder to convert it to powder form, and after that, it was sieved using 75 microns sieve. Physical properties like pore size and surface area were studied using scanning electron microscopy (SEM). Pollution removal studies were carried using the Jar test, and the experiments were designed using RSM. The results show that the maximum COD reduction of 98.58% was obtained when H₂SO₄ was used as an activation agent. The results show that the carbon activated by H₂SO₄ shows the highest removal than its counterparts. The optimum dose was optimized using RSM and found to be 300 mg/l, and the optimum reaction time was 10 min. By this set of conditions, 96.3% of COD removal could be achieved. The results are confirmed by SEM studies, which show a high surface area, more pores, and the presence of a high amount of carbon in the AC prepared using H₂SO₄.

Keywords Activated carbon · Date seed · Pollution removal · Response surface methodology · Waste to product

Introduction

Water pollution is the cause of great concern for modern society. Effluent discharged from industries contains various contaminants, which can cause environmental problems like eutrophication, algal bloom, and health threat to humans when released into the water bodies (Thamilselvi and Radha

2017). To mitigate adverse environmental effects, generally, conventional water treatment processes are employed to reduce the level of contaminants found in water. These methods include (but not limited to) biochemical processes, membrane filtration, coagulation, sedimentation, oxidation, adsorption by activated carbon (AC). Among these methods, the adsorption process by AC is widely used, especially after secondary treatment, and is well known to be useful for water and wastewater treatment (El-Sayed et al. 2014; Njoku and Hameed 2011; Thamilselvi and Radha 2017). Adsorption process by AC has several advantages over other methods. These advantages include less land area requirement for the AC; the adsorption process is not affected by toxic chemicals, and the AC is capable of reducing a high concentration of organic contaminants from water (El-Sayed et al. 2014). Activated carbons (ACs) are considered as the general adsorbent due to their broad range applications. These versatile materials are characterized by large specific surface areas and large pore volumes. Their principal property is to adsorb molecules as well in the liquid phase as in the gaseous phase

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(Sudaryanto et al. 2006). Activated carbon can be prepared from almost any organic material rich in carbon and preferably with low content in the inorganic matter (Li et al. 2008). There are plenty of reported studies for activated carbon preparation from a variety of agricultural waste such as date stones (Bouchelta et al. 2008; Haimour and Emeish 2006), peach stones (Soares Maia et al. 2010), rice straw (Uçar et al. 2009), pistachio-nut shells (Lua and Yang 2005), apricot stones (Şentorun-Shalaby et al. 2006), cherry stone (Olivares-Marín et al. 2006; Tancredi et al. 2004), pecan shell (Corcho-Corral et al. 2006), corncob (Deng et al. 2009), cotton stalk (Benadjemia et al. 2011), coffee bean husks (Zięzio et al. 2020), plain tobacco stems (Zięzio et al. 2020), olive bagasse (Saleem et al. 2019), and almond shells (Mohan et al. 2011). Different chemicals (KOH, K_2CO_3 , NaOH, Na_2CO_3 , $AlCl_3$, $ZnCl_2$, $MgCl_2$, H_3PO_4 , and H_2SO_4) are used as a chemical agent for the activation of carbons (Hadoun et al. 2013). Acid-activated pecan shell-based carbons show higher adsorption for organic matter than the commercially available activated carbon (Bansode et al. 2004). Specific properties of activated carbon depend on the raw material and the activation process employed (De Gisi et al. 2016). Several studies show that the activated carbon prepared at the lower temperature of carbonization exhibits better characteristics compared with activated carbon prepared at the higher temperature (Valizadeh et al. 2016; Islam et al. 2017; Norouzi et al. 2018; Liew et al. 2019). The chemical activation agent first degrades the cellulosic material, and the process of carbonization creates a suitable pore structure as a result of dehydration (Azevedo et al. 2007). Therefore, chemical activation affects the adsorption of activated carbon and ability based on differences properties such as surface area, density, pH, and conductivity (Puziy et al. 2002). Phosphoric acid imparts cation exchange capacity, making it chemically stable both in acidic and base media in addition to its thermal stability (Puziy et al. 2002). Oman is one of the largest producers of date. Date seeds are the waste material produced after processing dates and freely available, which makes them preferred material for activated carbon production in Oman. Though there are plenty of studies reported about activated carbon around the world, however, there is a shortage of studies in Oman. Also, there is a lack of data concerning the characteristics of the adsorbents such as their average particle size or specific surface area (De Gisi et al. 2016). Response surface methodology is a powerful tool to the optimization of process parameters, and it helps in selecting a set of the most suitable parameters in a given condition (Ghorbani et al. 2020; Karimifard and Alavi Moghaddam 2018; Topal and Arslan Topal 2020).

Moreover, there are not much-reported studies on the application of RSM for optimization of process parameters in adsorption study using activated carbon made from date seed using different activation agents. We reported

activated carbon production from coconut shells, orange peels, and banana peels using H_3PO_4 as an activation agent in our previous study (Shukla et al. 2020). The present study is aimed to ascertain the effects of varying activation agents on the pollution removal efficiency of date seed activated carbon using response surface methodology. In this study, response surface methodology was used to optimize different process parameters to achieve optimum removal. Physical properties of activated carbon were also studied using a scanning electron microscope.

Materials and methods

Date seeds were selected as raw material for the production of activated carbon because of its ample availability in Oman. Carbon was activated using three chemical activation agents HNO_3 , H_3PO_4 , and H_2SO_4 . Physical properties, like surface area and carbon content, were studied using an electron scanning microscope (SEM). Pollution removal experiments were designed by using response surface methodology (RSM) to optimize the process parameters for optimum removal. The detailed methodology is given below.

Preparation of activated carbons

Raw material date palm seeds were collected farm in Nizwa, Oman. First, the date palm seeds were washed with deionized water to remove impurities and dried in an oven at 110 °C for 24 h. The dry materials were then crushed. After that, the date seeds were carbonized in a muffle furnace at 300 °C for 1 h to produce carbon. Then, the produced material was cooled at room temperature for 30 min, then washed with distilled water, and then dried in the oven at 105 °C for 1 h. For chemical activation, the material was soaked into 1 N solution prepared from three different activation agents (H_3PO_4 , HNO_3 , and H_2SO_4) separately for 24 h and then again heated in the oven at 110 °C for 1 h. After that, the samples were heated in a muffle furnace at 300 °C for 1 h to achieve surface activation. After heating, the samples were cooled at room temperature. Then, the samples were soaked again in 100 ml of distilled water with 1 g of sodium bicarbonate for 24 h to remove excess acid from the sample. Samples were then washed separately with distilled water for 4–5 times until the pH becomes neutral. The washed samples were dried at 110 °C. The activated carbon of each material was milled and sieved to 75 µm, mesh size. Dried and sieved samples were stored in the clean and dry container for further experiments.

Physical properties of AC

The physical properties of AC like surface area and carbon content were studied using SEM at Central Analytical and Applied Research Unit, Sultan Qaboos University (CARRU/SQU). One sample was taken from activated carbon made using each activation agent (H_3PO_4 , HNO_3 , H_2SO_4) for the study.

Design of pollution removal experiment

Response surface methodology (RSM) is a statistical method that uses quantitative data from appropriate experiments to develop a regression model relating the experimental response to the process variables. A standard RSM design, central composite design (CCD) was used to design the number of experiments and to optimize the process conditions. The factors were the amount of activated carbon (100 mg/l, 200 mg/l, 300 mg/l) and time (10 min, 20 min, 30 min) with three different activation agents H_3PO_4 , HNO_3 , H_2SO_4 . The percentage of COD was taken as a response.

Pollution removal efficiency

Jar tests were conducted to evaluate the pollution removal efficiency of different activated carbons. Sixty-three samples were taken with different chemical activations and varying amounts of AC 100 mg/l, 200 mg/l, 300 mg/l as per the RSM design. The measured quantity of activated

carbon was added into the flask, which already contains 1-liter wastewater. After adding activated carbon, the sample was shaken at 120 rpm for different times as per the RSM design. After stirring, the samples were filtered using 40 number Whatman filter paper. Water quality parameters like pH and COD were analyzed using AWWA/APHA standard methods (APHA/AWWA/WEF 2005).

Results and discussion

Physical properties of activated carbon

Physical properties of activated carbon prepared using HNO_3

A scanning electron microscopy (SEM) technique was used to investigate the surface physical morphology of activated carbons. Scanning electron microscopic pictures of date seeds activated carbon produced by chemical activation using HNO_3 are presented. As shown in Fig. 1, there are channel-like walls on the surface of raw material and pores of different size and shapes also has different size of grains in the surface area; also, it shows the presence of various elements with a high amount of carbon around 75%. The external surface has a crack and a lot of grains particles with irregular and heterogeneous surface morphology with a well-developed porous structure.

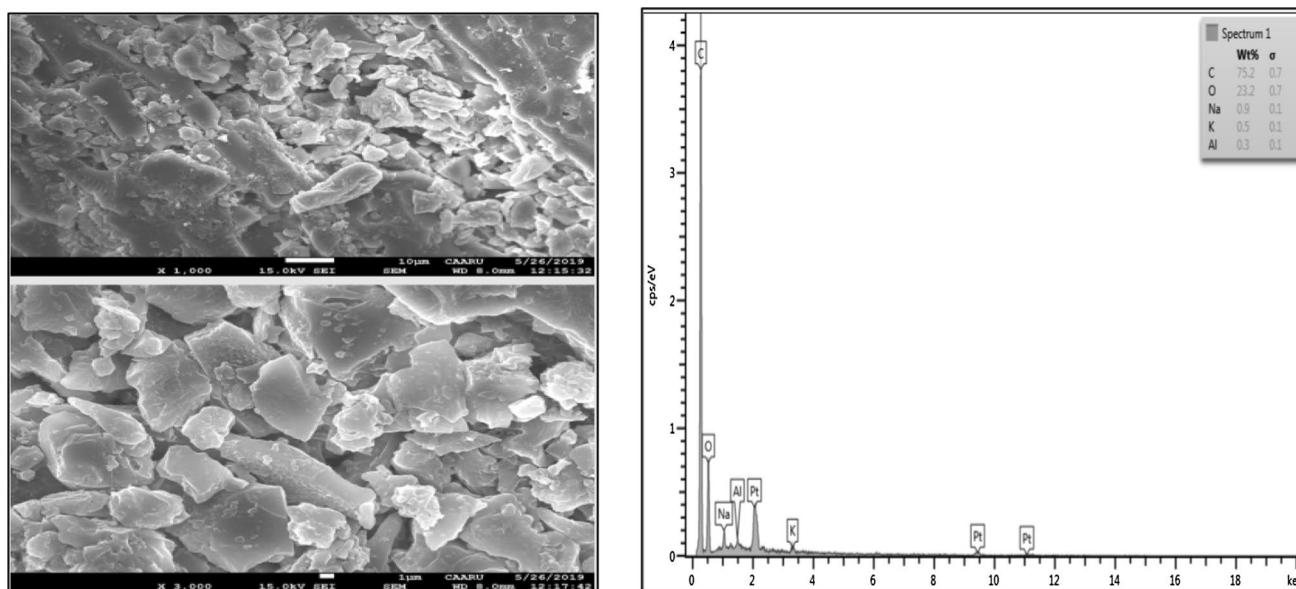


Fig. 1 Scanning electron microscopic analysis of activated carbon prepared using HNO_3 as an activation agent

Physical properties of activated carbon prepared using H_3PO_4

A scanning electron microscopy (SEM) technique was used to investigate the surface physical morphology of activated carbons. Figure 2 illustrates the SEM photographs of activated carbon prepared using H_3PO_4 . As shown in Fig. 2, the surface was smooth, and there was a channel-like wall on the surface of raw material and pores of different sizes, shapes, and small grains. The presence of various elements with a high amount of carbon (around 78.8%) can be seen.

Physical properties of activated carbon prepared using H_2SO_4

As shown in Fig. 3, activated carbon has a lot of pores with different sizes and shapes. As shown in the micrographs, the shape of the external surface of the activated carbons like a wall has cracks and some grains in various sizes in some holes and large numbers of voids. And the activated carbon morphology comes as a bone. The analysis of date seeds activated carbon activated by H_2SO_4 , shown in Fig. 3, showed the presence of various elements along with a high amount of carbon 82.6%.

Activated carbon made using different activation agents shows the significant formation of pores and rough surface and contains a high amount of carbon content. Moreover, activated carbon produced using H_2SO_4 shows more voids and 82.6% and carbon, which is highest among all. The

effects of the rough surface and high amount of carbon by this activated carbon were attributed to the high removal of COD during pollution removal studies.

Pollution removal efficiency

As per the RSM design, sixty-three samples were taken for different chemical activations and varying amounts of AC 100 mg/l, 200 mg/l, and 300 mg/l with different time (10 min, 20 min, and 30 min). The results obtained after the experiments are discussed below.

COD removal efficiency of activated carbon prepared using H_3PO_4

The relationship between different amounts of activated carbon, which are activated by H_3PO_4 and the time on-removal efficiency of COD, are presented in Fig. 4. The highest percentage of COD removal 95.74% was observed when the activated carbon is 200 mg/l and time is 30 min. Around 91.12% was the lowest removal at 10 min reaction time and 100 mg/l of activated carbon dose. A similar study was carried out by Mohammad Razi et al. (2018) with H_3PO_4 as an activation agent, and they observed 60% COD removal. It seems activation temperature plays a vital role as they activated at 550 °C; however, in the present study, we used a 300 °C temperature for activation.

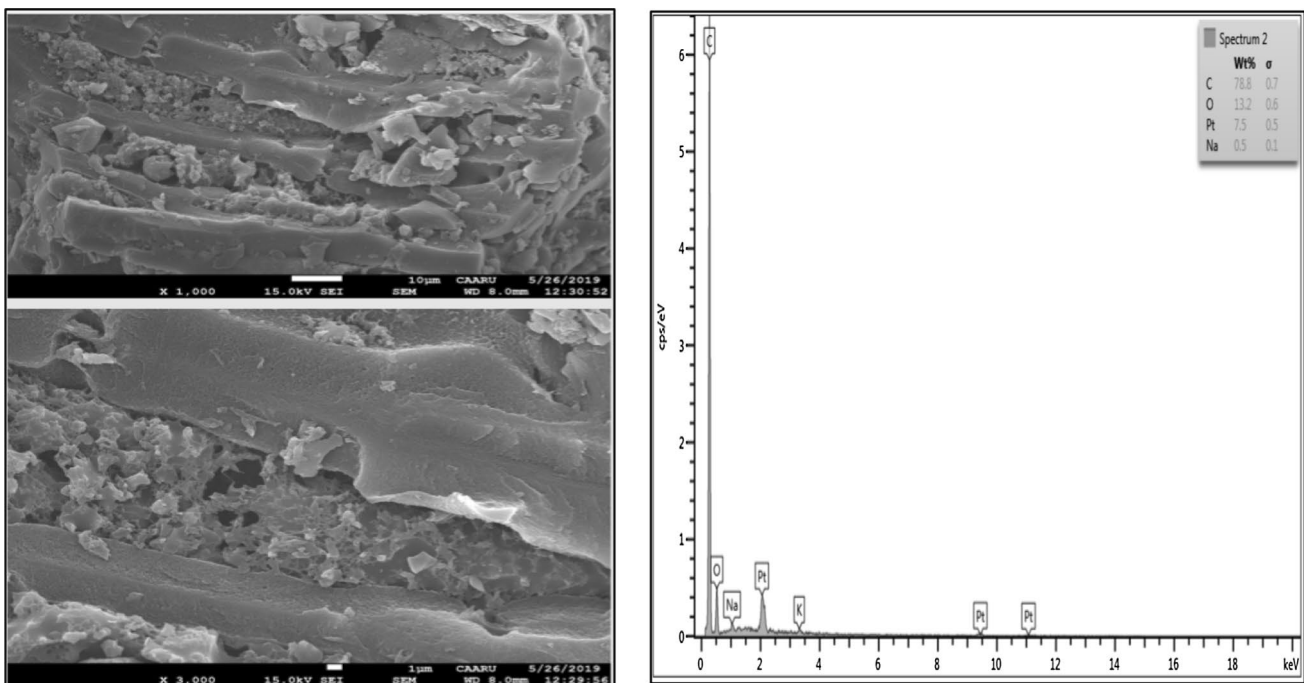


Fig. 2 Scanning electron microscopic analysis of activated carbon prepared using H_3PO_4 as an activation agent

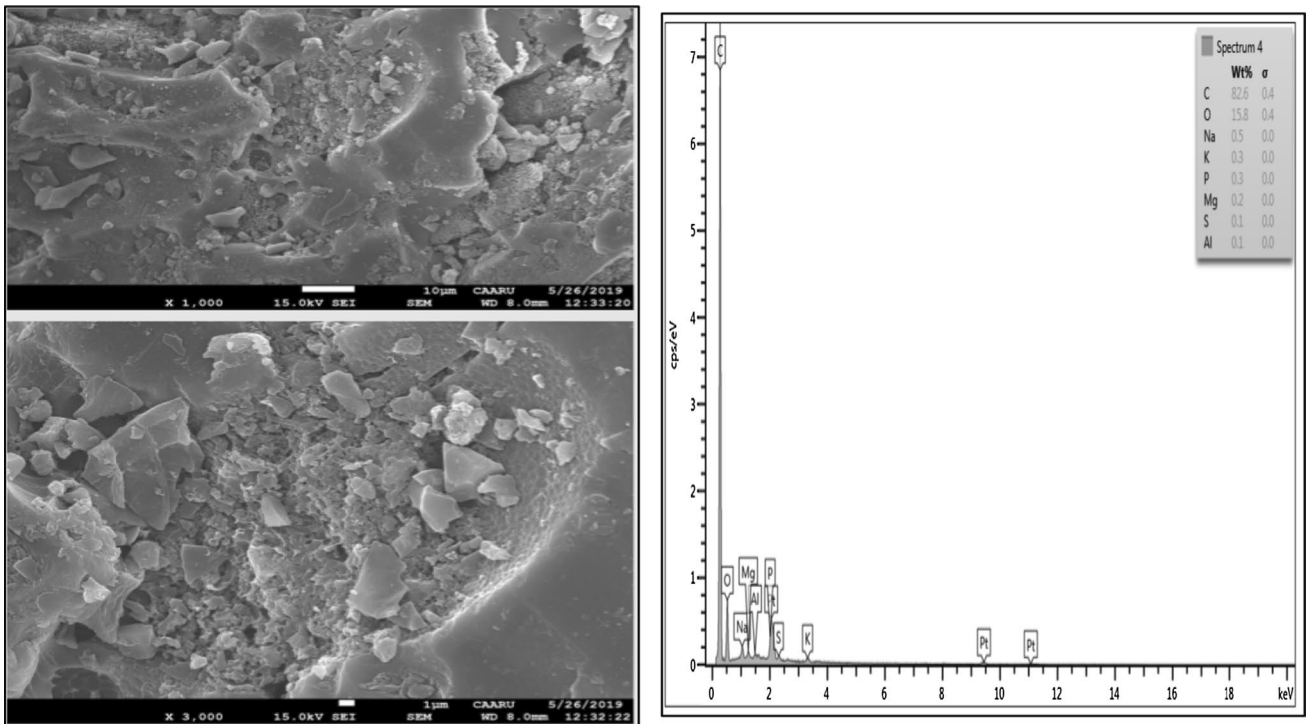


Fig. 3 Scanning electron microscopic analysis of activated carbon prepared using H₂SO₄ as an activation agent

Design-Expert® Software
 Trial Version
 Factor Coding: Actual

COD (%)

● Design points above predicted value

○ Design points below predicted value

88.4547 98.579

X1 = A: Amount of Activated Carbon
 X2 = B: Time

Actual Factor

C: Activated Agents = H₃PO₄

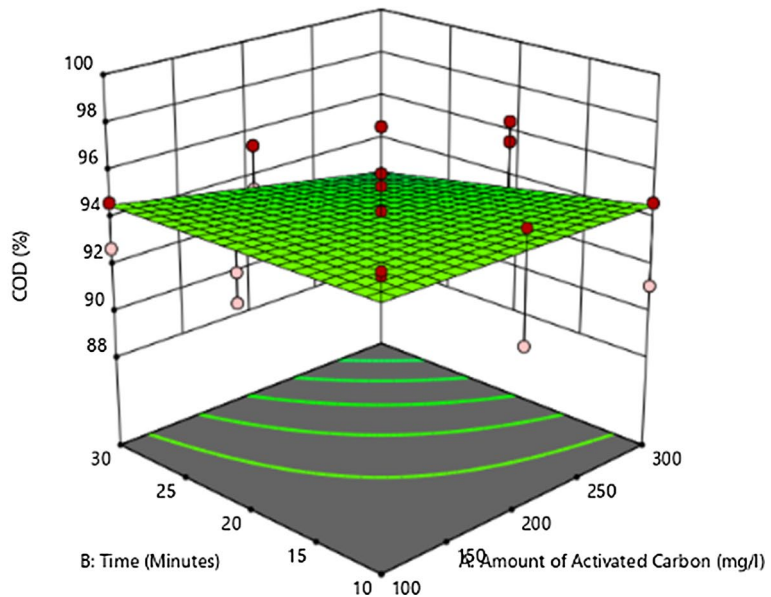


Fig. 4 Composite RSM graph showing the effects of various process parameters on COD removal efficiency of activated carbon prepared using H₃PO₄ as an activation agent

COD removal efficiency of activated carbon prepared using HNO_3

The effects of different amounts of activated carbon and different reaction times on COD removal efficiency for the activated carbon made using HNO_3 are presented in Fig. 5. The removal efficiency was found to be between 96.45 and 88.45%; it was found that the COD removal efficiency increases with an increase in the reaction time and amount of activated carbon. Maximum COD removal was observed at 30 min, and 300 mg/l activated carbon dose. Minimum COD removal was observed at 10 min reaction time and 100 mg/l dose. In a study, Yakubu et al. (2008) reported 91% removal of COD by the date seed activated carbon prepared using ZCl_2 as an activation agent; however, in the present study, 96% of maximum COD removal was observed. It indicates that HNO_3 is the stronger activation agent than ZCl_2 , which is also reported in the literature (Ademiluyi and David-West 2012).

COD removal efficiency of activated carbon prepared using H_2SO_4

The effects of different amounts of activated carbon and different reaction times on COD removal efficiency for the activated carbon prepared using H_2SO_4 are presented in Fig. 6. As shown in the figure maximum, 98.58% COD removal was observed at 30 min reaction time, and 300 mg/l activated

carbon dose. Minimum 91.65% COD removal was found at 10 min reaction time, and 100 mg/l activated carbon dose. The activated carbon prepared using H_2SO_4 shows better removal than its counterparts in this study, which might be because H_2SO_4 is a strong acid that could have been contributed to more activation, showing more voids (Fig. 3).

Optimization of process parameters

The set of optimized process parameters for the best result using response surface methodology is shown in Fig. 7. These sets of parameters can be selected to achieve high removal of COD with utilization less time and resources. As shown in Fig. 7, H_2SO_4 appeared to be the best activation agents among its counterparts in the study. The optimum dose was found to be 300 mg/l, and the optimum reaction time was 10 min. By this set of conditions, 96.3% of COD removal could be achieved. The desirability of this set is said to be 0.769, which is good, and this is one of the 31 combinations given by RSM.

Conclusion

It can be concluded that the removal of pollutants increases with the increase in the time and concentration of AC. The surface area of activated carbon was found to be large and has lots of pores of different sizes and shapes, and presence

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Factor Coding: Actual

COD (%)

● Design points above predicted value

○ Design points below predicted value

88.4547  98.579

X1 = A: Amount of Activated Carbon
X2 = B: Time

Actual Factor

C: Activated Agents = HNO_3

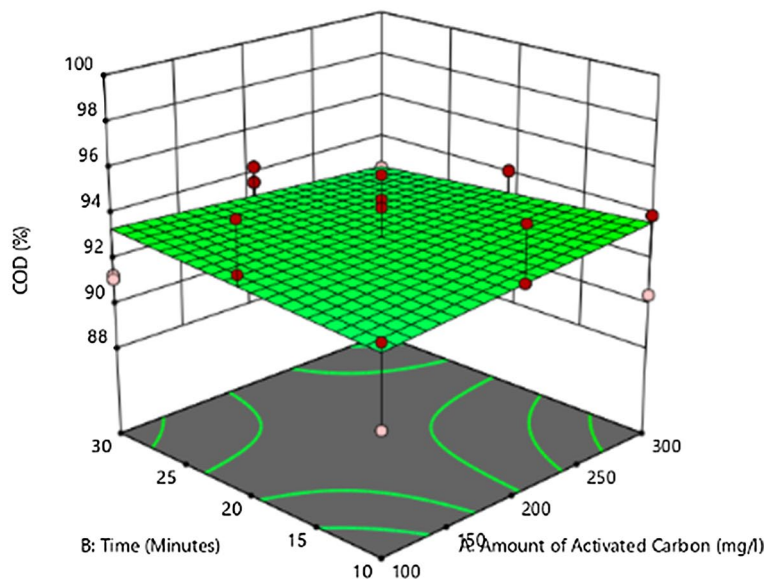


Fig. 5 Composite RSM graph showing the effects of various process parameters on COD removal efficiency of activated carbon prepared using HNO_3 as an activation agent

Design Expert® Software
 Trial Version
 Factor Coding: Actual

COD (%)
 ● Design points above predicted value
 ○ Design points below predicted value
 88.4547  98.579

X1 = A: Amount of Activated Carbon
 X2 = B: Time

Actual Factor
 C: Activated Agents = H2SO4

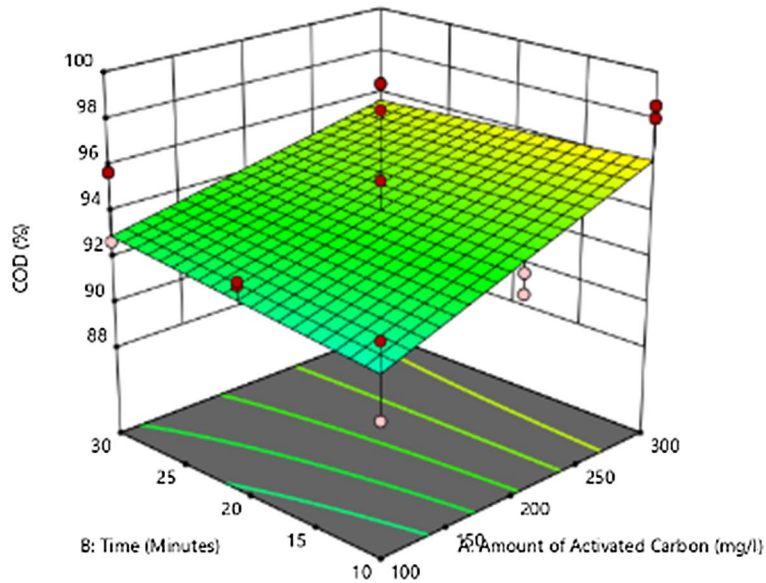
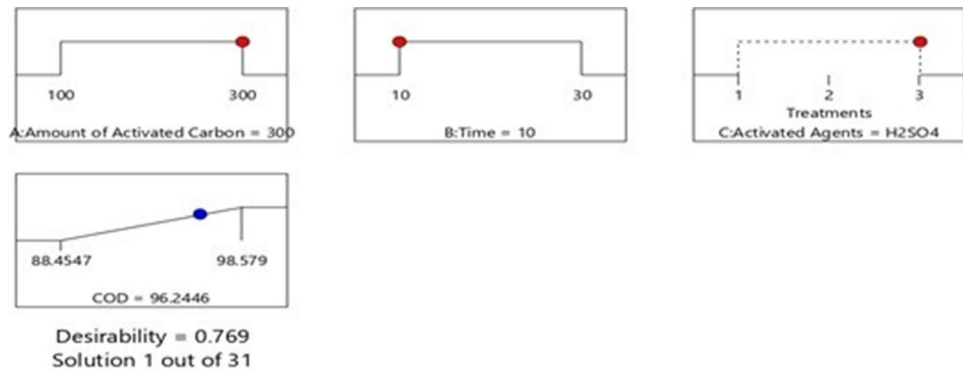


Fig. 6 Composite RSM graph showing the effects of various process parameters on COD removal efficiency of activated carbon prepared using H₂SO₄ as an activation agent

Fig. 7 Set of the optimized process parameters for the optimum removal of COD developed using RSM



of various elements with a high amount of carbon was also observed in the activated carbon prepared using H₂SO₄ activation. Among the three activation agents, H₂SO₄, HNO₃, and H₃PO₄, the activated carbons prepared using H₂SO₄ show better results in terms of pollution removal, which may be due to better activation occurred in case of H₂SO₄ acid as it is considered stronger acid than other two. The maximum COD removal of 98.58% was obtained. Optimization results show that 96.3% COD removal can be achieved by using 300 mg/l activated carbon prepared using H₂SO₄ acid at 10 min reaction time. It can also be concluded that eco-friendly, cost-effective activated carbon can produce using date seed. The use of low-cost adsorbents material to produce activated carbon for wastewater

treatment will be an alternative to the expensive method (commercial activated carbon like coal and wood). And by using organic waste to make low-cost activated carbon will reduce the load of organic waste in the landfill. By using response surface methodology, process parameters can be optimized to achieve desirable pollution removal at optimum resource and time.

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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