Perchlorate captured by activated carbon derived from dates seed through adsorption technique

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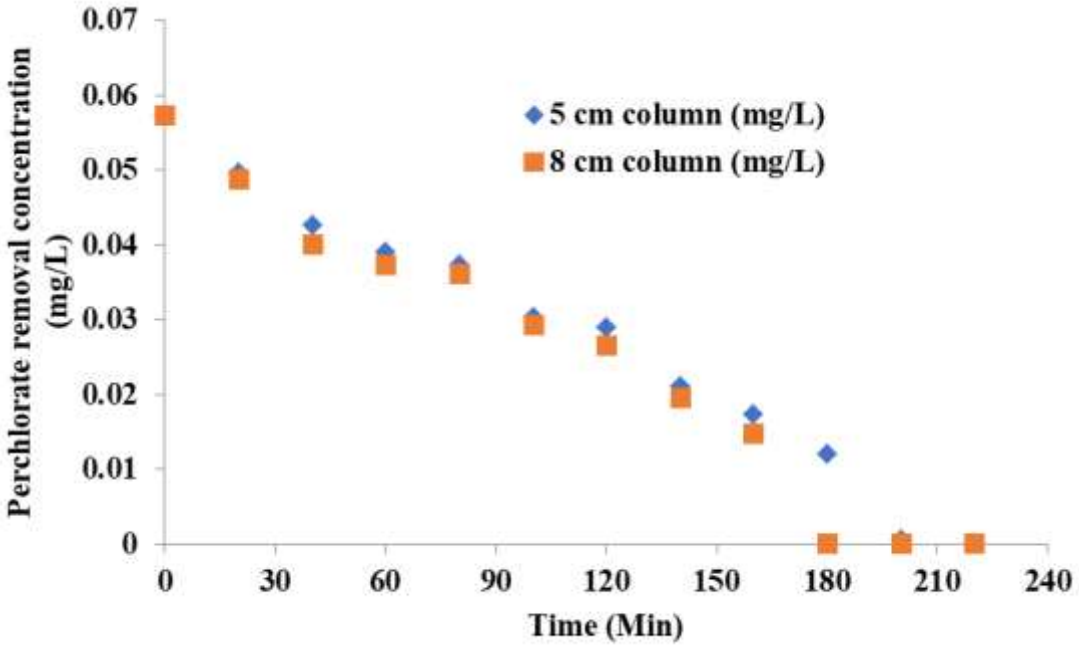
GRAPHICAL ABSTRACT



Abstract

In the present research packed bed column with activated carbon which is derived from the natural available dates seed used for exclusion of perchlorate from aqueous sample. The effect of several variables on adsorption performance has been studied, including inlet ion

concentration, flow rate, bed height, and temperature.The adsorbent was prepared in two different temperature 400℃ and 800℃ and the depth of column was 5cm and 8cm. The findings showed that the activated column at 400℃ in the depth of 5cm was saturated at 320 min and for 8cm column it is saturated at 300 min. Likewise for 800℃ columns in the depth of 5cm the treatment efficiency attained at 220 min and for 8cm column it was 180 min. It is showed that the optimal flow rate and bed height increase adsorption effectiveness. Gas chromatography (GC) was used to determine the perchlorate concentration in liquid sample after treatment process. The Thomas and Thomson mathematical models were utilized to determine the column's adsorption efficacy, with the maximum R^2value of 0.985 for 800°C in an 8 cm column with a removal efficiency of 98.1%. The surface morphology was examined with SEM, FTIR and XRD which proves that the pore changes in the morphology of the adsorbent created homogeneous to heterogeneous formation structure. This comprehensive study shown that activated carbon generated from dates seed is a successful adsorbent for the removal of perchlorate at optimal temperature and bed depth.

Keywords: Perchlorate, Chromatography, column study, surface morphology, regeneration

1. Introduction

A perchlorate is a chemical substance that contains the perchlorate ion, ClO4. Most perchlorates are salts that are manufactured commercially [1]. They primarily function as propellants, taking use of their potent oxidizing agents and ability to reduce static electricity in food packaging [2]. Primarily used as oxidizers in rocket, fireworks, and road flare propellants. A tetrahedral arrangement of oxygen atoms surrounds the chlorine atom in the heart of the perchlorate anion (ClO^{4-}). The species is a potent oxidizer since the oxidation state of chlorine is +7. It is widely utilized in the pyrotechnics business, as well as in some weapons and the production of matches [3]. Flammable source of combustible substance, such as black powder, saltpeter (an old name for potassium nitrate), or smokeless powder, such as cellulose nitrate, to produce energy. [4]. In water, potassium perchlorate salts dissolve and separate into the perchlorate anion and the salt's cation. Because it is the oxidizing component of flash powder, it is used in firecrackers [5]. A manufactured good that is typically produced for sale is a firecracker. Flash powder and a fuse enclosed in densely packed paper make up firecrackers [6]. By combining aluminum with perchlorate, flash powder is created. While Aluminum is either created using recycled Aluminum or extracted from the Earth's crust [7]. Due in part to its naturally occurring creation in the atmosphere

and deposition during rain or snow events, perchlorate has a nearly universal incidence in water in the nanogram consistent with litre variety [8]. Perchlorate has been measured in groundwater and surface water using incredibly precise methods. Perchlorate is also thought to occur often in seawater. Perchlorate concentrations in rainwater in China have recently been shown to range between 0.35 and 27.3mg/L, with a median value of 6.37mg/L [9]. A Chinese observer from Harbin, however, confirmed there were no detectable levels of perchlorate in the rain. Aspects of health effect the thyroid gland is a significant target organ for perchlorate poisoning in human [10]. It has been demonstrated that perchlorate partially prevents the thyroid from absorbing iodine. The creation of thyroid hormone requires iodine as a component [11]. When thyroid hormones are released into the blood, they modify positive frame characteristics. Although it hasn't been proven in people, it is hypothesized that long-term exposure to high levels of perchlorate in humans may also result in decreased thyroid hormone production [12]. The ability health effects of human exposures during the period from conception to adulthood at the age of 18 are covered in this section [13]. Children and growing fetuses may be more vulnerable to the effects of perchlorate than adults because they require thyroid hormones for optimal growth and development. water resources as a result of the natural resources of perchlorate [14]. The biodegradability and physico-chemical characteristics of pollutants have a significant impact on where they end up in the aquatic environment [15]. The lower half of the polarity-volatility diagram is where most of the aquatic contaminants that have been examined up to this point are located [16]. The highly polar and hydrophilic pollutants in the upper right quadrant of the polarity-volatility diagram are of enhanced relevance regarding the possibility of infiltrating drinking water resources due to their high mobility in water [17]. Perchlorate is a carcinogenic chemical that is harmful to both the environment and human health. The removal of perchlorate is a critical issue [18]. For the purpose of removing percholrate from a sample of groundwater, the adsorption procedure is used. Adsorption occurs when residual or imbalanced forces are present at the bottom of a liquid or solid phase [19]. The molecular species that it comes into touch with on the floor tends to be drawn to it and maintained by these uneven residual forces [20]. Adsorption primarily occurs on floors. Adsorption is a phrase that is distinct from absorption since it primarily occurs at the substance's surface, whereas absorption involves a uniform dispersion of the material throughout most persons. Sorption is the method used when both adsorption and absorption occur at the same time [21]. Adsorbent and adsorbate are additives used in the adsorption process [22]. The substance on which adsorption occurs is known as the adsorbent. The material that is being adsorbed at the ground is called

adsorbate [23]. The major goal of this study is to ascertain the dates seeds' ability to adsorb perchlorate from water samples. study the effectiveness of the adsorption process about various bed depths, concentrations, and temperatures, as well as the effectiveness of the adsorption process regarding various bed depths, concentrations, and to analyze the breakthrough profiles for the sorption.

2. Materials and methods

2.1 Preparation of Column Material

The naturally occurring dates seed was harvested, dried completely for 24 hours in a high air oven at 100°C, and then manually broken into many pieces [24]. It was then thoroughly ground to create granular activated carbon. To get rid of the microbiological particles, the carbon material was three times washed with sodium chloride solution [25]. The carbon material was heated in order to activate it. The adsorbent materials prepared in two different temperature 400℃ and 800℃ [26]. The sodium perchlorate used in this study was created by oxidizing ammonium chloride to perchlorate, doubly decomposing with sodium chloride solution at 100°C, and then cooling the reaction mixture to 10°C [27]. For the preparation of perchlorate solution analytical standards, deionized water is employed.

2.2 Column study

A glass column with an interior diameter of 2 cm and a length of 60 cm was utilized in the fixed bed column studies. Activated carbon with particle sizes ranging from 0.6 mm to 2 mm was used [28]. The bottom of the column was covered with glass wool and filled with activated carbon. The 5cm and 8cm high beds were used. The employed flow rates ranged from 2.5mL/min [29]. After taking samples at predefined intervals, IC assessed the residual perchlorate concentration in the effluent samples. When the column neared exhaustion, column studies came to an end [30]. Column investigations are carried out at room temperature for practical reasons.

2.3 Gas chromatography

After an experimental method involving adsorption, the perchlorate level in the sample was determined using gas chromatography [31]. The anion stock standard solutions for per chlorate were taken, and the water utilized met the standards of ISO 3696, Grade 1. Perchlorate solution from several sources was used for method validation and calibration trials, as well as an independent source for quality control [32]. The gas chromatographic

system included a gradient pump with a flow rate of 0.25 mL/min, an auto sampler with a 10µL injection loop, a column thermostat set at 30°C, an eluent generator supplying 35 mmol/L KOH, and a conductivity detector. [33]. The suppression column was a Dionex AERS, and the column was a Carbon 18 with guard column, both in 2-mm format. Each sample ran for a total of 30 minutes [34]. Statistics were used to assess the working range, performance, and calibration data for a concentration decade investigating standard perchlorate solutions [35].

2.4 Kinetic study

2.4.1. Thomas Model

The Thomas model was used to calculate the liquid attentiveness dependency with time [36]. Incessant column technique took inner and outside mass transfer limitations into account.

$$\ln\left(\frac{\mathrm{Co}}{\mathrm{Ct}}-1\right)=(\mathrm{Kt}*q*m/Q)-kt*Co*t$$

2.4.2. Thomson Model

The Thomson model was used to uncover the relationship between the interior and exterior adsorption processes. The Thomson model can be expressed as

$$ln\left(\frac{Co}{Ct}-1\right)= k_{TH}\, q_e W - k_{TH}\, C_o\, t$$

2.5 Regeneration of adsorbent

Thermal regeneration was carried out by electrically heating of adsorbent at 350°C in both an inert and an environment of air [37]. In the hot water extraction process, the adsorbent was heated in a muffle furnace to 200℃ to 400℃, twice washed with DI water and NAOH solution, and then dried for another 5 hours in a hot air oven. By assessing the perchlorate (100 mg/L) removal capability, the regeneration effectiveness of the regenerated adsorbent was ascertained.

2.6 Characterization of adsorbent

2.6.1. SEM Analysis

The surface morphology images of the adsorbent materials were characterized using the SEM instrument. The instrument was run using its default settings, which are frequently used to scan samples' surfaces for the finest photos at several exaggerations of 10,000 and 20,000 times. The SEM limitations utilized in this investigation espoused and matched suitably.

2.6.2. XRD pattern

XRD spectroscopy was used to examine the d-spacing utilizing LD anode material with J-Alpha1 wavelength of 2.655, J-Alpha3 wavelength of 2.844, and J-Beta wavelength of 3.481, XRD was used to evaluate the d-arrangement of dense ingredients and to determine the crystallinity and parallel plane distance (d-spacing) of adsorbent materials. The approach was used precisely.

2.6.3. FTIR Analysis

The molecular structure of the adsorbent materials was confirmed by an FTIR spectrometer operating in default mode, which is commonly used to image tasters with PC-based software that controls utensil process and statistics dispensation. For FTIR analysis, a modest quantity of adsorbent materials was created. Data on infrared transmittance were gathered spanning the wave number series of 6000 cm^{-1} to 800 cm^{-1}. Adsorbent was used as a blank throughout the analysis of all samples [38]. The spectrum data were compared with a reference to determine the practical clusters and kind of covalent bindings vibration present in the sample.

3. Result and Discussion

3.1 Adsorption study

Perchlorate adsorption was investigated using continuous process studies. Every twenty minutes, a sample was taken for the perchlorate removal investigation, and GC was utilized to measure the effectiveness of treatment [39]. The first round of treatment research used column study that were 5 cm and 8 cm in diameter packed column in a muffle furnace to 400°C for two hours. In 5 cm column the influent capacity was 2.5 mL/min and the effluent range was 0.5 mL/min. The influent range for 8cm column is same as 5cm column and the effluent range was 0.42 mL/min. In both different sized columns, the concentrated of perchlorate used in this treatment process was 0.0573 mg/L. For 5 cm in 320 minutes the column got saturated and for 8 cm in 300 minutes, the column was completely saturated.

Table.1 Perchlorate removal study for 400°C adsorbent

Time (min)	5 cm column (mg/L)	8 cm column (mg/L)
0	0.0573	0.0573
20	0.0536	0.0451
40	0.0487	0.0386
60	0.0421	0.0335
80	0.0374	0.0324
100	0.0320	0.0250
120	0.0300	0.0200
140	0.0254	0.0194
160	0.0211	0.0187
180	0.0161	0.0169
200	0.0128	0.0098
220	0.0101	0.0061
240	0.0082	0.0042
260	0.0060	0.0040
280	0.0021	0.0031
300	0.0014	0.0001
320	0.0001	0.0001

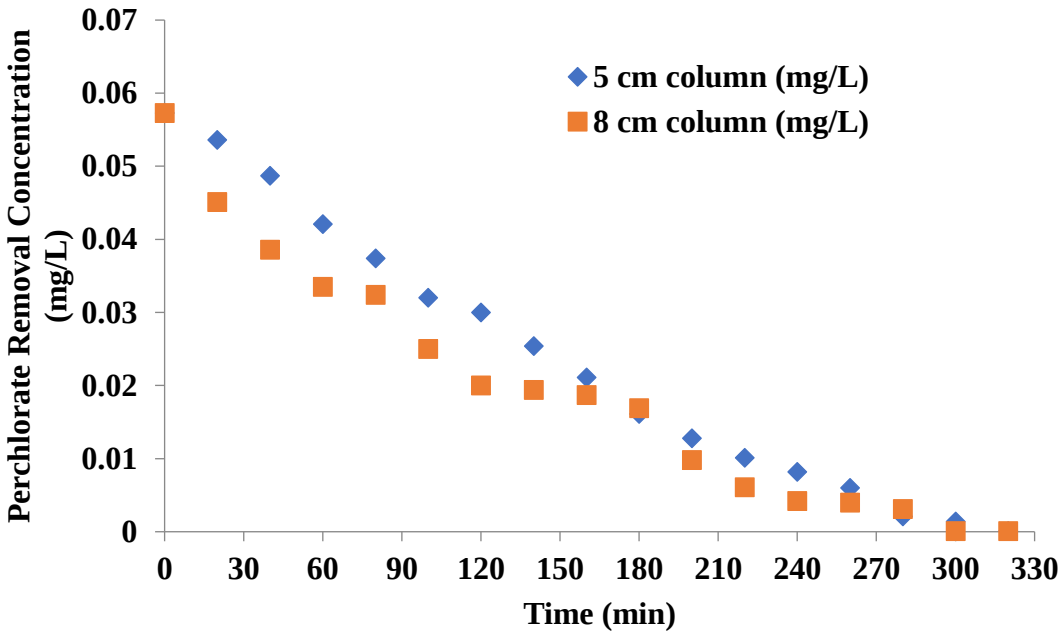


Figure.1 Perchlorate removal by 400°C adsorbent

For the second series of column studies, two different sized columns were used to evaluate the removal of perchlorate. The sample had been collected every 20 minutes, and GC utilized to determine the effectiveness of treatment [40]. The initial round of treatment studies was carried out using 5 cm and 8 cm columns heated in a muffle furnace for three hours at 800℃. The output ranges were 0.5 mL/min and 0.42 mL/min, although the ingestion trial level 2.5 mL/min in 5 cm and 8 cm. The initial treatment effectiveness began at 0.0573 mg/L. For 5

cm the column depleted at 220mins and in 8 cm the column was completely depleted in 180 minutes.

Table.2 Perchlorate removal study for 800°C adsorbent

Time (min)	5 cm column (mg/L)	8 cm column (mg/L)
0	0.0573	0.0573
20	0.0496	0.0489
40	0.0427	0.0401
60	0.0391	0.0374
80	0.0373	0.0362
100	0.0304	0.0294
120	0.0290	0.0266
140	0.0212	0.0196
160	0.0174	0.0149
180	0.0121	0.0001
200	0.0007	0.0001
220	0.0001	0.0001

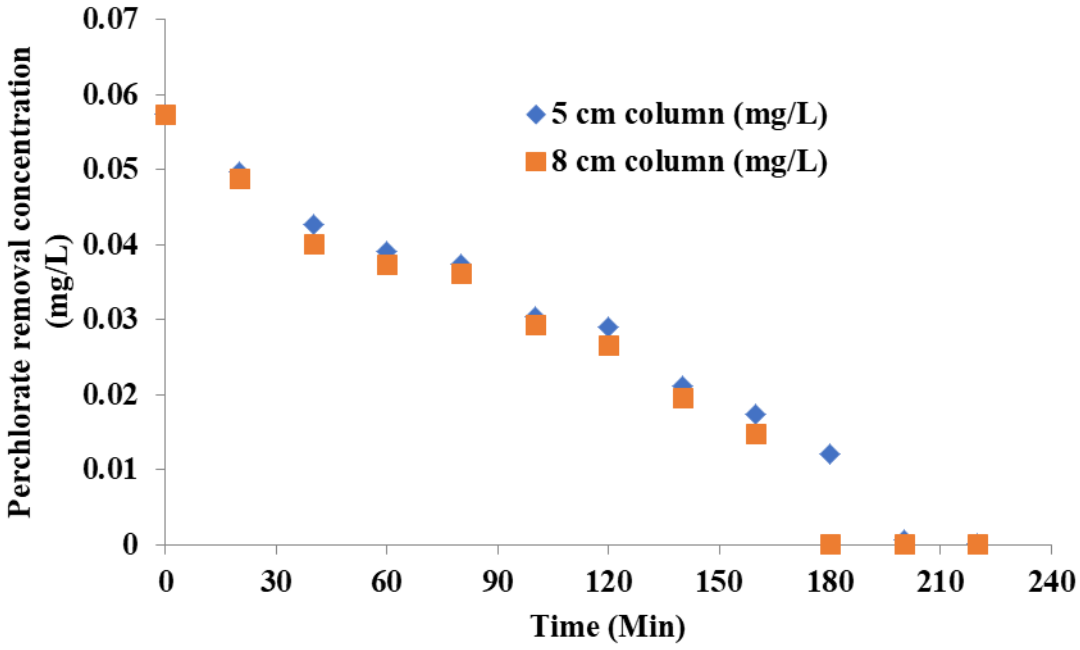


Figure.2 Perchlorate removal by 800°C adsorbent

3.2 Determination of perchlorate by Gas chromatography Technique

In order to examine the chromatogram, the samples were collected after the adsorption study it was preserved in refrigerator. A sample of 10 µl inserted into consumption region and passed through the movable and motionless phases. when GC output graph was

used to estimate the optimal perchlorate level. The graphical depiction was analysed by software, and gas chromatography was used to determine the perchlorate level [41]. Based on the height and area of the peak obtained in the graphical depiction, the level of perchlorate content in the sample was calculated.

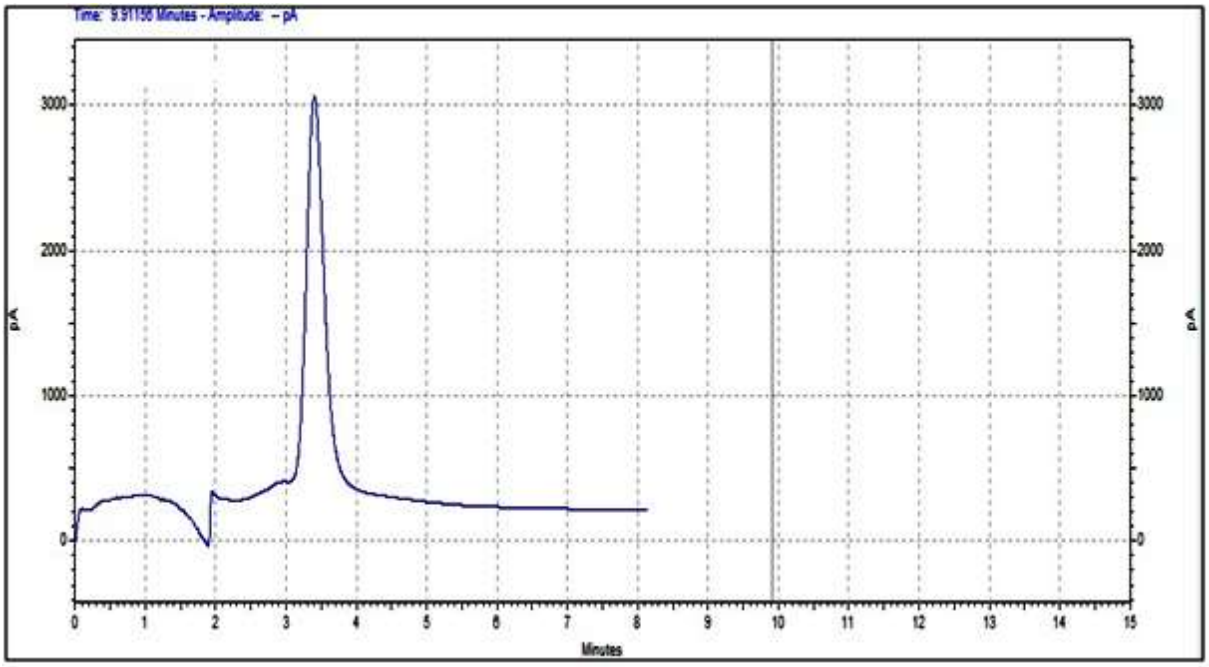


Figure.3 Perchlorate range obtained by Gas Chromatography

3.3 Kinetic Study

3.3.1. Thomas Model for adsorption study

The adsorbate's ionic speciation in the binding site oversaw directing the adsorption process. The Thomas model used to evaluate it. The unrushed and anticipated adsorption process break through curves were consistent, resulting in the greatest regression coefficient value (R^2) of 0.985 for an 8cm column at 800ºC. The effect of mass (m) and concentration time (t) on kt's value (t) suggested that adsorption kinetics were favourable at the maximum concentration of adsorbate. Initially, kt values were in a lowered condition, and in the following level, kt (ml/min.mg) values climbed as 0.015, 0.029 for 400ºc column and it is dropped from 0.011and 0.006 for 800ºc column for 0.0573 mg/L concentration [42]. The rate constant (kt) dropped with increasing temperature. There was a small rise in the equilibrium uptake capacity (qo). The decline in qo demonstrated that the adsorption capacity is inversely correlated with bed height and contact time.

Table.3 Thomas Model for two sized column

Material	Column size (cm)	k_t x 10^{-3} (mL/(min.mg)	q_o (mg/g)	R^2
Dates Seed 400°C	5	0.015	19.08	0.910
	8	0.029	0.40	0.929
Dates Seed 800°C	5	0.011	14.49	0.943
	8	0.006	0.008	0.985

3.3.2. Thomson model for adsorption study

The results of the experimental study were outstanding for the Thomson model. The input concentration (Co) was found to be uniformly maintained in the 2.5ml/min range. The alteration in kTH value was similarly influenced by bed height [43]. The k_{TH} decreased from 0.072 to 0.008 mL/min.mg at 0.0573 mg/L with heated adsorbate material at 400°C for a 5cm and an 8cm column. The q_e increased from11.50 to 18.87mg/g. The R^2 range for 5cm column was 0.954 and for 8cm column it was 0.929 in the temperature range of 400ºc. The Thomson model for the 800ºc column revealed that for a concentration of 0.0573 mg/L with heated adsorbate material at 800°C for a 5 cm column and an 8cm column, k_{TH} increased from 0.38 to 0.450 mL/min.mg.The q_e depleted from 0.987 to 0.42mg/g. The R^2 range for 5cm column was 0.966 and for 8cm column it was 0.984 in the temperature range of 800ºc.

Table.4 Thomson model for Dates 400°C

Column Depth	5cm	8cm
qe (mg/g)	11.50	18.87
k_{TH} (mL/min.mg)	0.072	0.008
R^2	0.954	0.929

Table.5 Thomson model for Dates 800°C

Column Depth	5cm	8cm
qe (mg/g)	0.987	0.42
k_{TH} (mL/min.mg)	0.38	0.450
R^2	0.966	0.984

The Thomson and Thomas model was used in mathematical modelling to assess the adsorption studies. Both studies were tested with two different column sizes, and they fit the model well. The two processes for 8cm column in the temperature 800ºc the R^2 values crossed 0.98, indicating a well-fit association for the adsorption process. Highly contaminated samples have been collected for an adsorption investigation to measure the treatment efficacy. The infiltration capacity of the adsorbent has been impacted by the 800°c thermal heating [44]. Characterization of adsorbents is the next significant phenomenon. On quantitative analysis is the main emphasis.

3.4 Regeneration of Adsorption Column

The regenerated column supported three cycles of adsorption process. In each cycle the column slowly losing the removal capacity. For regeneration process the temperature used between 200–400°C. The 55% of perchlorate was leached into the DI water during regeneration of column [45]. Sodium chloride and HCl were used for chemical regeneration. Both regeneration agents displayed excellent regenerative effectiveness of 90%. The foremost downside of using sodium chloride as a regeneration mediator is that it necessitates an additional step to remove excess sodium ions from the material before further modifying it with HCl. The use of HCl as the regeneration agent, however, eliminates the need for this extra step [46]. As a result, HCl is the best regenerative agent for treating adsorbent and making it reusable with minimal effort.

3.5 Characterisation of adsorbent

3.5.1. SEM Analysis Adsorbent

SEM performed for examination of adsorbent's surface morphology, and the images are shown in Figure 4. This illustration demonstrates how adsorbent particles, which range in size from 200 nm to 1.62nm, are combined at random and built into derived adsorbent constituents. This shows that the adsorbent particles have a size range of around 686.6 nm to 1414.3 nm. Even though adsorbent particles with a size of 686.6 nm or smaller were not found. SEM clearly shows that they exist. In order to visualize the pores as being nanoscale in size, Additionally the photos of the various pores can be expanded, allowing us to see the numerous turns and basins that were formed at random on the corresponding pores [47]. These facts show that the perchlorate molecules contained in the manufactured adsorbent materials show a substantial protagonist in the creation turns and basins through the comprehensive construction of carbon-carbon bonds.

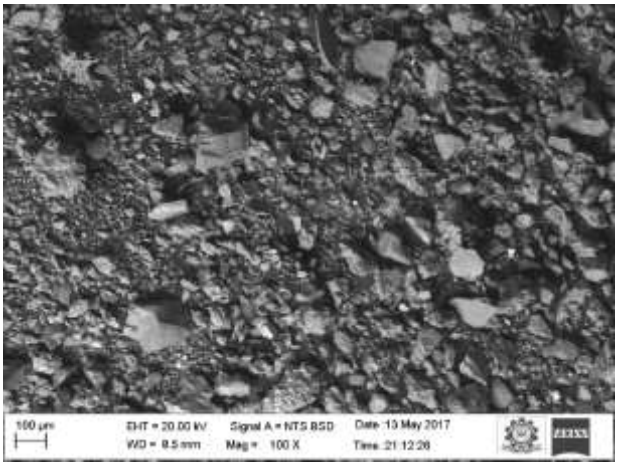


Figure.4 SEM of adsorbent

3.5.2. XRD pattern of adsorbent

The diffractograms pattern displayed in figure.5 an indicated the presence of three atomic plane clusters, which are typical nanostructures of manufactured adsorbent materials. Two-dimensional X-ray diffractograms of the 8º–10º, 30º–40º, and 50º–60º ranges were provided by the first cluster, the 30º–40º, 86º–10º, and the 50º–60º by the second and third clusters, respectively, with maximum intensities of about 98, 86, and 10 a.u, respectively, and 10 a.u, respectively, by the third cluster [48]. The qualified number of atomic clusters represented by the strength of the diffractogram peaks; it is generally known that each value denotes the separation between atoms that are arranged in parallel planes. Due to this circumstance, micro porous nanostructures within the particles were appropriately produced and connected to one another in a variety of forms and sizes. Additionally, the fundamental framework of the created molecular nanostructures for adsorbent materials is more fully depicted in figure 5.

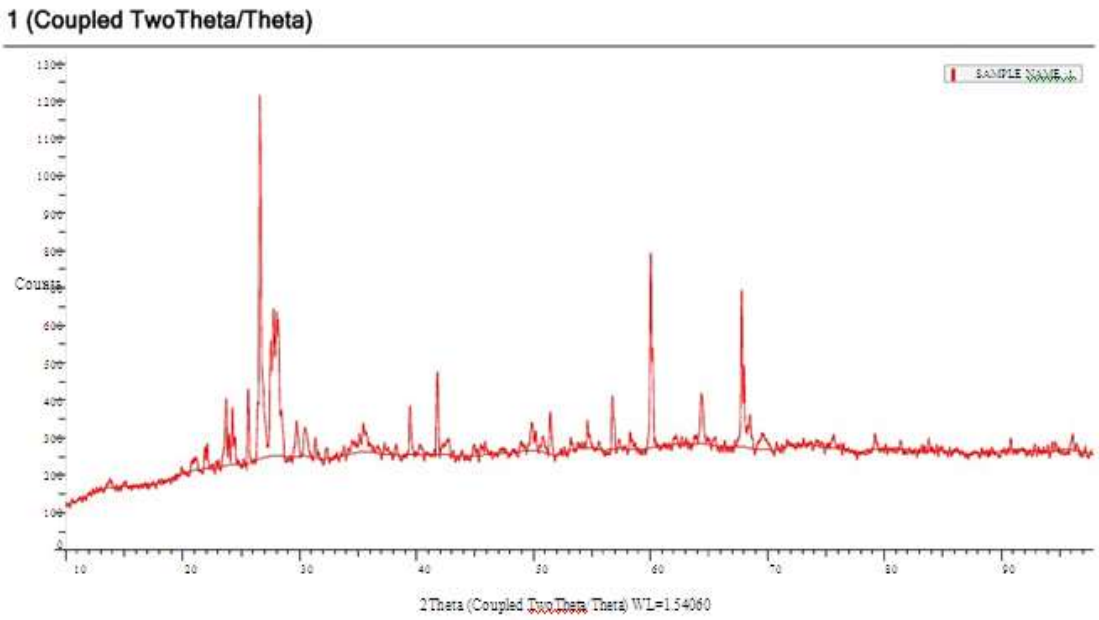


Figure.5 XRD pattern of adsorbent

3.5.3. FTIR Analysis of adsorbent

Figure.6 displays the FTIR graphical range produce adsorbent materials. This picture demonstrates adsorbent elements engendered by interpretable FTIR spectra crests, with wavenumbers of 4800 cm^{-1}, 3700 cm^{-1}, 3200-1200 cm^{-1}, 1000-600 cm^{-1}, and 860 750 cm^{-1} [49]. These spectra indicated that the manufactured adsorbent material's molecular framework corresponds to several functional groups. Since a tiny wave number peak with an apparent wavelength of roughly 4700 cm^{1} and a nonbonded O-H stretching vibration was seen, these are water molecules that can be trapped by relevant material's pores or in sheets with a graphene-like structure. [50]. C-H out-of-plane bend, trans C-H out-of-plane bend, and cis C-H out-of-plane bend are the others. There also methyl (=CH-), methylene (=CH2), alkenyl C=C, and skeletal C-C. These atomic bond vibrations demonstrated that the produced adsorbent material is mostly composed of molecular structures like graphene. This structure closely resembles that found in XRD analyses.

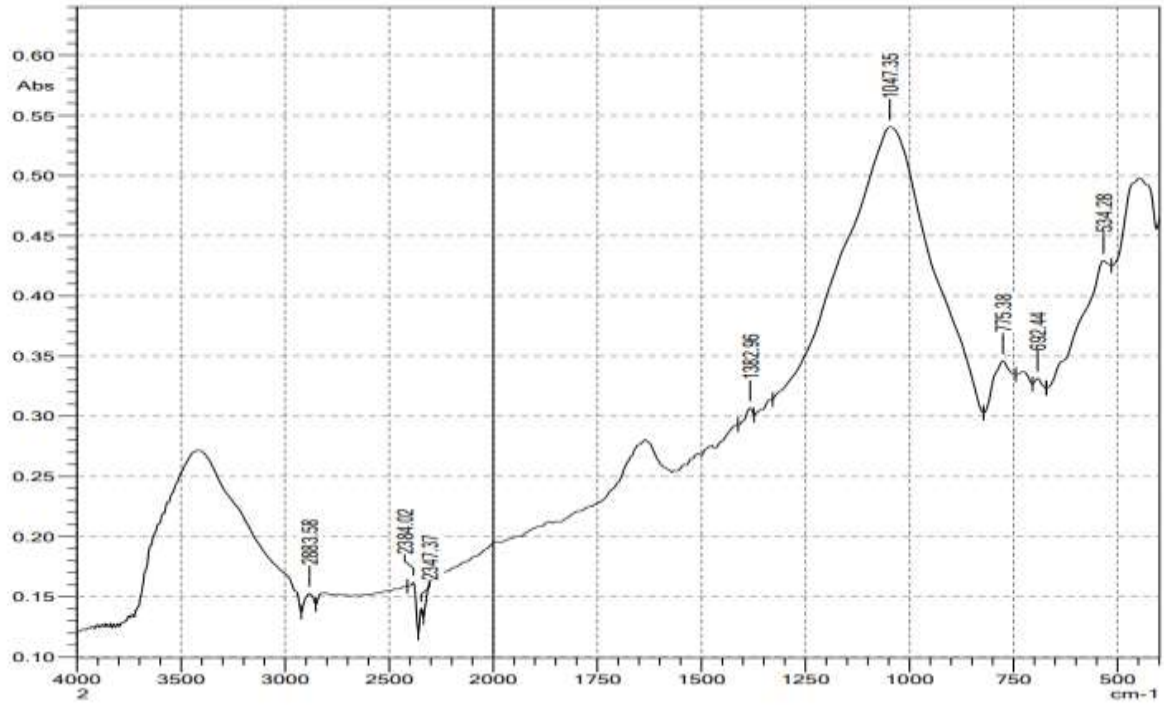


Figure.6 FTIR spectrum of adsorbent

4. Conclusion

It has been established that confiscating perchlorate from water with an adsorbent derived from dates seed works well and is affordable. According to results, Perchlorate could be removed from water using modified activated carbon. In a continuous bed column investigation, process variables including bed height, initial perchlorate concentration and flow rate significantly affect adsorption of perchlorate. Minor perchlorate inlet attentiveness, higher adsorbent bed height and slower feed flow were discovered to increase the continuous bed column adsorption system's performance. By comparing R^2 values and breakthrough curves, both the Thomas and Thomson models can be used to characterize the behaviour of perchlorate adsorption in a continuous column investigation. The experimental data and the Thomas and Thomson models agreed well. Even in low perchlorate concentration, the adsorption continued and the removal efficiency attain with the adsorbent. This study proved that increasing bed height with higher temperature in the optimum flow rate achieved the better removal of perchlorate in the aqueous solution. The SEM, XRD, and FTIR analysis showed that the perchlorate adsorption process had slightly altered the surface morphology of the adsorbent. The investigation of regeneration was done using thermal and chemical approaches. Chemical regeneration is the most efficient approach which proved based on easy of operation and depends on regeneration method.

Conflict of Interest

The authors declares no conflict of interest

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