Zinc chloride modification of sage leaves powder and its application as an adsorbent for KMnO₄ removal from aqueous solutions

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Abstract

Sage Leaves Powder was modified by 30% w/w of Zink Chloride (ZnCl₂) and used as a cheap adsorbent for potassium permanganate (KMnO₄) adsorption from aqueous media. The characterization of the synthesized adsorbent showed diversity in its functional groups at 1030, 1700, and 2930 cm⁻¹. The impact of experimental conditions on the adsorption was investigated. It was found that these experimental conditions except pH have positive effects on the efficiency of this adsorbent. The initial KMnO₄ concentration of 1500 mg l⁻¹, the temperature of 60 °C and the solution pH of 1.5 were found to be the ideal conditions in this adsorption. The equilibrium data were analyzed using three isotherm models. Langmuir model was found to be the best model applied in this work. The adsorption capacities were 714.29, 769.23, 833.33 and 909.09 (mg g⁻¹) at 30, 40, 50 and 60 °C, respectively. The parameters of the adsorption kinetics were evaluated by three kinetic models. The kinetic results confirm that the 2nd order described well the experimental data. Thermodynamic outcomes suggest that this adsorption is a spontaneous and endothermic process. The obtained results make from this adsorbent a good option for KMnO₄ removal from wastewater in terms of its low-cost and adsorption performance.

1. Introduction

The best and most chemical agent has been used for the reduction and elimination of hazardous chemical pollutants from water, reservoir, and underground wells are KMnO₄ [11]. Notably, it was found that KMnO₄ has the ability to remove taste and odor from water, and it stops the bacterial growth of iron bacteria [1]. It has also been used as an oxidizing agent to get rid of some inorganic species such as Fe, As and Mn from groundwater [2, 3], it also mainly used to oxidize many phenols, dyes, cyanide, and organic compounds [4–8], where KMnO₄ enters a series of oxidation and reduction reactions within different mechanisms, including electron and oxygen transfer [9]. Moreover, water purification with KMnO₄ was adopted instead of chlorination [9]. Unfortunately, extreme intake of manganese may lead to damage to dopaminergic neurons and a lot of acute health problems like skin and eye irritations, while chronic health effects of KMnO₄ summarized in risk on the liver and kidneys [10, 11]. Moreover, it was reported previously that the Manganese has higher toxicity towards Daphnia Magna [12] and leachate contains Manganese has superior toxicity on Vicia faba micronucleus [13].

The excess quantities of KMnO₄ remain in tap and ground waters after water treatment processes by using of this agent (KMnO₄). The higher concentrations of KMnO₄ in the treated water transform the watercolor into the distinctive pink color of this dye and sometimes to the brown color that is never desirable. Therefore, the disposal of KMnO₄ from treated water became the subject of most researcher’s interest.

Many chemical and physical methods have been developed to eliminate KMnO₄ from aqueous solutions, among them the adsorption technique was widely acclaimed [1]. As it is often considered an easy way to prepare and use, moreover, it does not produce secondary outcomes as in some other methods [1]. One of the most famous adsorbents used to remove KMnO₄ is activated carbon, due to its high effectiveness in adsorption of KMnO₄ from contaminated water [14].

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Activated carbon-based on coconut shells have been used to eliminate of KMnO₄ from aqueous solutions [14]. Adsorption technology was also applied using activated carbon prepared from corn cob and bone [15], activated charcoal, and the activated carbon modified by sulfuric acid also have been applied [1, 16]. Despite the high efficiency of activated carbon against organic and inorganic pollutants, the cost of manufacturing is very high.

Recently, metallic oxides nanoparticles NPs were also used to treat wastewater as active adsorbents for various organic and inorganic dyes [17–23]. However, over time, it turned out to be a very expensive adsorbent. This is the reason that prompted many to search for new adsorbents that depend on their preparation from low-cost materials. Lately, *Nitraria retusa* leaves and *Neem* leaves powders have also applied for KMnO₄ removal from aqueous samples [24, 25].

*Salvia officinalis* (*Lamiaceae*) is known by *sage* herb in many countries, it has been widely used as traditional medicine due to impressive medicinal properties with no side effects, the higher constants of volatile oils give it curative power [26]. Sage herb leaves have been used in flavoring tea and food, manufacturing of modern medicine, cosmetics, and fragrance [27]. Sage (*Salvia officinalis, Lamiaceae*) has also been used in folk medicine in many countries for a long time ago to treat colic, cold symptoms, and rheumatism [28]. Moreover, it used as a nerve cradle and to stimulates blood circulation [28].

Up to this time, many studies and researches have been performed to find out new adsorbents with high efficiency and reasonable cost to adsorb the dyes from the polluted water. Until now, no material based on *sage* leaves has been used in any form to rid the water from dyes even KMnO₄, despite the wide diffusion of this plant, in addition to its amazing healing properties.

Therefore, the main aim of this research is to synthesis a new adsorbent from *sage* herb and to assess the performance of this adsorbent with regard to removing KMnO₄ from contaminated water. Isotherms, kinetics, and thermodynamics parameters will also be investigated. In addition, factors that influence the behavior of this adsorption will also be examined.

2. Materials and methodology

2.1. Materials

Chemicals such as KMnO₄ with the purity of 99.00%, ZnCl₂ with the purity of 98.00%, hydrochloric acid (HCl) with a concentration of 37.00%, Sodium hydroxide (NaOH) with the purity of 98.00%, and Sodium carbonate (Na₂CO₃) with a purity of 99.50% were obtained from Sigma-Aldrich and used in this work.

2.2. Adsorbent modification

Dry sage leaves were obtained from the local markets of Tabuk, Saudi Arabia, and it was ground using mortar. A 0.5 l of 30% w/w of ZnCl₂ was mixed with a 130 g of sage leaves powder. Then this mixture was refluxed for 120 min, after that the hot mixture was left for cooling at room temperature following by filtration. The residual solid was boiled with 0.5 l of 1 M HCl for 60 min to get rid of ZnCl₂ from the sample. The obtained powder was sieved to achieve particles of the same size.

2.3. Characterization of adsorbent

The solution pH at which the adsorbent surface is uncharged (pHZPC) value for this adsorbent was assessed according to Theydan and Ahmed method [29]. In order to estimate the density, porosity, and surface area of the synthesized adsorbent surface, the analyzer technique was used (NOVA-2200 Ver. 6.11). Whereas the morphology of the adsorbent and the surface functional groups were identified by using Microscopy of Scanning Electron (SEM) and Spectroscopy of Fourier Transform Infrared (FTIR) (Nicolet iS5 of Thermo Scientific FT-IR), respectively.

2.4. Experiment for the pH solution impact

A batch adsorption experiment was achieved to investigate the impact of pH solution on KMnO₄ adsorption from aqueous solution. 20 ml of the 700 mg l⁻¹ KMnO₄ solution with various pH values (1.5–11) were added to six amber bottles having 0.04 g of the solid adsorbent. pH was adjusted for each by using 1 M HCl or 1 M NaOH solution. Then bottles were shaken in an automatic shaker for 20 h at 30 °C temperature and 160 rpm. filtration was done for each sample followed by measuring of KMnO₄ concentration using a 6800UV-visible spectrophotometer (Jenway) at λmax 525 nm. Finally, the adsorption amounts at equilibrium were determined by applying equation (1).
\[ q_e = \frac{(C_i - C_e)V}{m} \quad (1) \]

\( q_e \) (mg g\(^{-1}\)); equilibrium adsorption amount, \( C_i \), KMnO\(_4\) primary concentration (mg l\(^{-1}\)); \( C_e \), equilibrium concentration of KMnO\(_4\) (mg l\(^{-1}\)), \( V \), KMnO\(_4\) solution volume (L), \( m \), adsorbent mass (g).

### 2.5. Equilibrium and thermodynamic experiments

Equilibrium experiments have been taken placed to study the impacts of temperature and KMnO\(_4\) primary concentration on the adsorption process and to determine the isotherm and thermodynamic parameters of this adsorption. In these experiments, 20 ml of KMnO\(_4\) solutions with various concentrations (300–1500 mg l\(^{-1}\)) have been mixed with 0.04 g of the synthesized adsorbent in 25 ml amber bottles. The shaking process was performed for each mixture for 20 h at four different temperatures (30, 40, 50, 60 °C) and 160 rpm. Then the immediate filtration and UV-visible measurements were achieved to determine the concentration of non-adsorbed KMnO\(_4\) in the filtrate. KMnO\(_4\) adsorption amounts \( q_e \) were evaluated by applying equation (1). Finally, the calculated \( q_e \) (mg g\(^{-1}\)) values were plotted against initial concentration \( C_i \) (mg g\(^{-1}\)) for each temperature to study the effects of temperature and concentration of KMnO\(_4\) on the performance of this adsorption.

Moreover, the obtained results from this part have been analyzed by the isotherm models represented in equations (2), (3) (Langmuir), (4) (Freundlich), and (5) (Temkin).

\[ \frac{C_e}{q_e} = \frac{1}{q_{max} K_L} + \frac{C_e}{q_{max}} \quad (2) \]

\[ R_L = \frac{1}{1 + K_L C_i} \quad (3) \]

\[ \ln q_e = \ln K_F + \frac{1}{n} \ln C_e \quad (4) \]

\[ q_e = B_1 \ln K_T + B_1 \ln C_e \quad (5) \]

Where the maximum adsorption capacity (mg g\(^{-1}\)) is \( q_{max} \), the dimensionless factor is \( R_L \), constants of Langmuir, Freundlich, and Temkin equations are \( K_L \), \( K_F \), and \( K_T \), respectively. The intensity of the adsorption constant is \( n \) and a constant related to adsorption heat is \( B_1 \).

Whereas thermodynamic parameters (\( \Delta H^o \), \( \Delta S^o \), \( \Delta G^o \)) of this adsorption were estimated using equations (6) and (7).

\[ \ln \left( \frac{q_e}{C_e} \right) = -\frac{\Delta H^o}{RT} + \frac{\Delta S^o}{R} \quad (6) \]

\[ \Delta G^o = \Delta H^o - T \Delta S^o \quad (7) \]

Since, the free energy, entropy, and enthalpy changes are \( \Delta G^o \), \( \Delta S^o \), and \( \Delta H^o \), respectively. T and R are the temperature (K) and general gas constant (0.0083 314 KJ K\(^{-1}\) mol\(^{-1}\)).

### 2.6. Kinetic experiments

In this section, 20 ml of 300, 400, and 500 mg l\(^{-1}\) of KMnO\(_4\) solutions have been shacked for 5 min with 0.04 g of the prepared adsorbent at a rotation speed of 160 rpm and 30 °C. Then the filtration was carried out. After that residual KMnO\(_4\) concentrations in the filtrate have been detected by using UV/Visible spectrometer at \( \lambda = 525 \) nm. Equation (8) was applied for the calculation of the adsorption quantities \( q_e \).

\[ q_e = \frac{(C_i - C_e)V}{m} \quad (8) \]

A \( q_e \) (mg g\(^{-1}\)) refers to the removed amount of KMnO\(_4\) at any time. \( C_i \) and \( C_e \) (mg l\(^{-1}\)) represent KMnO\(_4\) concentrations at primary and time \( t \), respectively. \( V \) and \( m \) are the solution volume and adsorbent mass.

To examine the impact of agitation time on KMnO\(_4\) adsorption and to determine kinetic parameters, similar experiments were repeated at various period times ranged from 5 to 960 min.

The linear expressions for the 1st order (equation (9)), 2nd order (equation (10)) and intra-particle distribution (equation (11)) have been employed for evaluating the parameters of this adsorption kinetics.

\[ \log(q_e - q_i) = \log q_e - K_i \frac{t}{2.303} \quad (9) \]

\[ \frac{t}{q_e} = \frac{1}{K_2 q_{max}^2} + \frac{t}{q_e} \quad (10) \]

\[ \frac{t}{q_e} = \frac{1}{K_1 q_{max}^2} + \frac{t}{q_e} \quad (11) \]
where the equilibrium adsorption amount and the amount of KMnO₄ removed from the solution after each time intervals are \( q_e \) and \( q_t \), respectively. A kinetic parameter related to the boundary-layer width is \( C \). Whereas, the constants of 1st order, 2nd order, and intra-particle distribution models are \( K_1 \), \( K_2 \), and \( K_{diff} \), correspondingly.

3. Results and discussion

3.1. Adsorbent properties

The results obtained from the physisorption of nitrogen gas (N₂) on chemical modified Sage leaves powder showed that total pore volume for all pores of a diameter smaller than 2809.424 Å, average pore diameter and the area of the adsorbent surface are 0.00464 cc g⁻¹, 2647.813 Å, and 104.78 m² g⁻¹, correspondingly. The SEM image for the modified Sage leaves powder (figure 1) proves that the adsorbent surface contains several asymmetrical macropores and bulky holes which has an important role in the behavior of adsorption. The randomness of macropores on the surface and the large variation in their size will allow the absorbent material to move very freely through the surface.

Figure 2 illustrates the relation between \( \text{pH}_i \) and \( \text{pH}_{f} \) and indicates that \( \text{pH}_{ZPC} = 6.2 \). Since, at solution pH less and higher than 6.2, the adsorbent surface will be positively and negatively charged, respectively. Similar results have reported previously by Al-Aoh [25]. The spectrum of FT-IR for this adsorbent was demonstrated in figure 3. This figure shows that there are three peaks of absorption in the regions of 1030, 1700, and 2930 cm⁻¹. These absorption bands (at 1030, 1700, and 2930 cm⁻¹) are correlated to ring vibrations, ketones, and C–H stretching, correspondingly [30, 31].

This great diversity of chemical functional groups present on this prepared adsorbent surface will characteristically affect the adsorption process. As it is often the adsorption on the surface occurs between the particles of the adsorbate and the adsorbent surface functional groups.

3.2. pH solution impact

The most important that factor affects the adsorption is the pH solution, as it was reported that the adsorbate ionization degree and the adsorbent surface charge depend on the value of pH solution [32].

To figure out the adsorption performance through a large range of pH (1.5–11), the amounts of permanganate adsorbed (\( q_e \) mg g⁻¹) are plotted against pH values as it is demonstrated in figure 4. A slightly and sharply decreasing in the adsorption amount can be observed from figure 4 when the solution pH raised in the ranges of (1.5–6.2) and (6.2–11), correspondingly.
Adsorption at pH less than 6.2 was due to the force of attraction between MnO$_4^-$ and the positively charged adsorbent surface. The positive charges on the adsorbent surface are decreased with increasing pH value from 1.5 to 6.2. This is considered the real reason for the slight decrease in the values of $q_e$ when pH elevated from 1.5 to 6.2. Whereas, the adsorbent surface will be negatively charged at pH solution over 6.2 and the force of repulsion between the negative charges of the adsorbent surface and MnO$_4^-$ causes the sharply decreasing in the $q_e$ values when pH increased from 6.2 to 11.

3.3. Equilibrium and thermodynamic studies
Figure 5 illustrates the relationship between $q_e$ (mg g$^{-1}$) of this adsorption and adsorbate concentration at a temperature of 30, 40, 50, and 60 °C. This figure showed that the values of $q_e$ (mg g$^{-1}$) are increased and almost be constant when KMnO$_4$ concentration increased in the ranges of (300–1300 mg l$^{-1}$) and (1300–1500 mg l$^{-1}$),
correspondingly. The increment observed when the adsorbate concentration increases from 300 to 1300 mg l\(^{-1}\) was due to raising the force of dynamic, which reduces the mass movement resistances at the surface interface [33, 34]. Whereas, most adsorption active sites almost be saturated over 1300 mg l\(^{-1}\). Moreover, this figure confirms that this adsorption is positively affected by rising temperatures, which indicates the endothermic process. The endothermic process observed in this work resulted from decreasing and increasing the solution viscosity and adsorbate molecules kinetic energy correspondingly due to rising temperature [35]. Similar observations were reported previously for dyes adsorption by activated carbon based on low cost materials [25, 36, 37]. It was also observed by Ibis and Asoluka [38] that the adsorption of toxic metal ions by agro-waste is positively affected by temperature.

The isotherm parameters were achieved in this work from intercepts and slopes of the plots of \((C_e/\bar{q}_e)\) against \(C_e\), \((\ln\bar{q}_e)\) against \(\ln C_e\) and \((\bar{q}_e)\) against \(\ln C_e\) represented in figures 6(a)–(c) for Langmuir, Freundlich, and
Temkin, respectively. The values of isotherm parameters and $R^2$ values were listed in Table 1. In comparison between $R^2$ of these three models, it can be suggested that the obtained data of this adsorption follows the Langmuir model better than the other models used in this work. This proves that this adsorption is a monolayer and the adsorbent active sites are homogenous. Similar observations were reported by Legrouri et al.\cite{39} for adsorption of some heavy metals on the activated carbon prepared from molasses.

Slopes and intercepts of the plots of $\ln(\frac{q_e}{C_e})$ vs. $1/T$ (1/K) (Figure 7) have been used for calculating the thermodynamic parameters of this adsorption recorded in Table 2. The positive values of $\Delta H^\circ$ and $\Delta S^\circ$ (Table 2) confirm an endothermic process of KMnO$_4$ adsorption by this adsorbent\cite{40} and decreasing the randomness of this adsorption, respectively\cite{41}. These results are completely identical to the results of previous studies\cite{25}. Furthermore, decreasing the randomness at the interface by increasing KMnO$_4$ concentration is evidence that the initial adsorbate concentration considered the most effective variable affecting this adsorption. While $\Delta G^\circ$ values are negative (Table 2), indicating that this adsorption is a spontaneous process. The same results were described for KMnO$_4$ adsorption by neem leaves powder\cite{25} and adsorption of other dyes by polymer nanocomposites\cite{42}. Copper and Lead sorption by biomass of Fumaria indica was found to be spontaneous\cite{43}. Moreover, adsorption of divalent metal ions on the groundnut shell is spontaneous\cite{44}.

The adsorption capacities ($q_{\text{max}}$) obtained in this work were found to be 714.29, 769.23, 833.33 and 909.09 (mg g$^{-1}$) at 30, 40, 50, and 60 °C, correspondingly. These outcomes are evidence that this low-cost adsorbent used in the work will meet higher attention in water and wastewater purifications.
Table 1. Parameters values related to isotherms for KMnO₄ adsorption by sage leaves powder modified by ZnCl₂.

| Temperature | Langmuir | Freundlich | Temkin |
|-------------|----------|------------|--------|
|             | $q_{\text{max}}$ (mg g$^{-1}$) | $K_L$ (l mg$^{-1}$) | $R_L$ | $R^2$ | $K_F$ (mg g$^{-1}$) | $(l$ mg$^{-1})^{1/n}$ | $1/n$ | $n$ | $R^2$ | $K_T$ (l mg$^{-1}$) | $B_T$ | $R^2$ |
| 30 °C       | 714.29   | 0.00318    | 0.162 | 0.997 | 17.3578 | 0.5630 | 1.7762 | 0.988 | 0.0467 | 166.39 | 0.99 |
| 40 °C       | 769.23   | 0.00659    | 0.132 | 0.997 | 23.3571 | 0.5398 | 1.8525 | 0.987 | 0.0634 | 168.04 | 0.991 |
| 50 °C       | 833.33   | 0.00846    | 0.106 | 0.994 | 27.4290 | 0.5517 | 1.8126 | 0.986 | 0.0820 | 182.31 | 0.986 |
| 60 °C       | 909.09   | 0.01455    | 0.064 | 0.995 | 39.6662 | 0.5531 | 1.8080 | 0.977 | 0.1423 | 196.96 | 0.988 |
3.4. Comparison study

The great interest in the purification of water from all of the pollutant types attracted many researchers to develop and manufacture new effective and cheap adsorbents. Recently KMnO₄ was widely applied in various human activities and considered as a hazardous water pollutant. Thus, many different adsorbents were applied for the elimination of this dye from polluted water. Among them, modified sage leaves powder has been used as a new and cheap adsorbent in this work for KMnO₄ adsorption from aqueous media. The performance of the adsorbent used in this work and the other adsorbent previously used for adsorption of KMnO₄ from solutions were presented in table 3. The adsorption capacities summarized in this table confirm that the modified sage leaves powder is the best compared with the other adsorbents.

3.5. Adsorption kinetics

To examine the adsorption time influence, the shaking time (t) was plotted against qₜ (adsorption amount at any time t) for adsorption of three different KMnO₄ concentrations (300, 400, 500 mg L⁻¹) by a fixed amount of a chemical modified sage leaves powder (figure 8). Figure 8 showed that there are three different regions upon the contact time, fast KMnO₄ adsorption in the range of (5–45 min), then a slightly increasing in the adsorption rate upon extending the contact time from 45 to 420 min, and the third region is related to equilibrium state occurred after 420 min. Adsorption at the beginning of the time was very fast because all the adsorbent effective sites were empty, after 45 min the adsorption rate slows down as the active sites on the adsorbent surface begin to be filled by MnO₄⁻ ions and then the equilibrium obtained because there is no vacant adsorption active site available over 450 min. These outcomes are agreeing well with the results of other studies regard the permanganate elimination from water and wastewater [14, 24, 25]. Moreover, the experimental results of kinetic have been analyzed by the kinetics models of 1st order (figure 9), 2nd order (figure 10), and intra-particle-diffusion (figure 11). The parameters of the kinetics were calculated from the intercepts and slopes of these kinetic plots demonstrated in these three figures. The calculated parameter values summarized in tables 4 and 5. The linear relationships that can only be observed in figure 10, higher values of R² and the agreement between the calculated values of qₑ by applying the 2nd order kinetic model and experimental qₑ values (table 4) confirm that this adsorption followed 2nd order kinetic model. A similar observation was reported for KMnO₄.
adsorption by activated carbon [14]. Furthermore, adsorption of divalent metal ions by groundnut shell [44] and Uranium by modified Vigna radiata waste biomass [45] followed the 2nd order kinetic model.

Based on figure 11 and table 5, there are no good linear relationships between \( q_t \) and time (t) of the obtained two regions. Moreover, the plots are not pass through the original point. This confirms that the diffusion of the intra-particle step is not the limiting step in this adsorption. This means that the distribution and movement of KMnO4 particles through the pores on the surface of the sage will be extremely easy. This corresponds to what was indicated by the SEM results, as it was clear that the pores present on the surface of the sage are numerous and irregular.

### 4. Conclusions

The present work showed that Zinc Chloride modified sage leaves powder as a cheap adsorbent has a significant adsorption capability towards KMnO4. The achieved outcomes indicate that KMnO4 adsorption by chemical
modified sage leaves Powder is positively affected by increasing each temperature, initial concentration, and experimental time. Whereas, this adsorption is negatively affected by increasing solution pH. The data of the isotherms were analyzed by Langmuir, Freundlich, Temkin models and it was found that the better performed of data analysis was by the Langmuir isotherm model. The adsorption capacities were found to be 714.29, 769.23, 833.33, and 909.09 (mg g\(^{-1}\)) at 30, 40, 50, and 60 (°C), correspondingly.

The parameters of kinetics were estimated graphically, the obtained \(R^2\) values are along with calculated and experimental KMnO\(_4\) amounts removed are strongly assured that the KMnO\(_4\) adsorption by modified of sage leaves powder follows the 2nd order kinetic model. The Thermodynamic outcomes designated that this adsorption is endothermic and spontaneously occurring. The higher adsorption capacities and rates of the sage leave powder observed in this work for KMnO\(_4\) absorption, its availability and low-cost, ease of preparation, and

![Figure 9](image1.png)

**Figure 9.** Pseudo 1st for KMnO\(_4\) adsorption by sage leaves powder modified by ZnCl\(_2\).

![Figure 10](image2.png)

**Figure 10.** Pseudo 2nd order for KMnO\(_4\) adsorption by sage leaves powder modified by ZnCl\(_2\).
use will attract attention and highlights more for using sage leaves powder for adsorption of KMnO4 from aqueous media in the coming future.

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