



## Short Communication

# A new insight to the physical interpretation of activated carbon and iron doped carbon material: Sorption affinity towards organic dye



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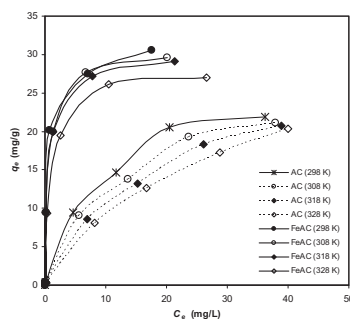
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## HIGHLIGHTS

- Successful iron incorporation onto the porous carbon surface was achieved.
- The significance of impregnation is revealed from the enhanced potential of FeAC.
- Above 95% methylene blue removal was observed by using FeAC.
- MB uptake was more feasible at ambient temperature.

## GRAPHICAL ABSTRACT



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## ABSTRACT

To enhance the potential of activated carbon (AC), iron incorporation into the AC surface was examined in the present investigations. Iron doped activated carbon (FeAC) material was synthesized and characterized by using surface area analysis, energy dispersive X-ray (EDX), temperature programmed reduction (TPR) and temperature programmed desorption (TPD). The surface area of FeAC (543 m<sup>2</sup>/g) was found to be lower than AC (1043 m<sup>2</sup>/g) as a result of the pores widening due to diffusion of iron particles into the porous AC. Iron uploading on AC surface was confirmed through EDX analysis, showing up to 13.75 wt.% iron on FeAC surface. TPR and TPD profiles revealed the presence of more active sites on FeAC surface. FeAC have shown up to 98% methylene blue (MB) removal from the aqueous media. Thermodynamic parameters indicated the spontaneous and exothermic nature of the sorption processes.

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## 1. Introduction

Activated carbon (AC) materials are very well known for their versatile surface characteristics. The variety of pore size and pore volume, high specific surface areas, low cost, extended sorption capacity, surface functional groups and thermal stability make the use of AC as a better choice for its application in water and wastewater treatment worldwide (Paul and Kim, 2009). However, modification of the surface of the as received activated carbon to

improve their performance when applied to industrial effluents and other water systems, is receiving more attention (Chen et al., 2013). Usually wet or dry oxidation processes are applied to modify the surfaces of raw materials. In the wet oxidation method, the surface of AC is treated with an oxidizing agent solution.

Dyes are visible pollutants of water systems. They are frequently added into water systems as waste water from industries such as leather, textile, plastics, food, rubber paper and cosmetics, etc. (Pandey et al., 2007). Methylene blue (MB) is a basic dye widely used in the above mentioned industrial applications. The uncontrolled release of dye residues as industrial effluents into the water streams is increasing water pollution at large. The health

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hazards associated with the dyes contaminations include jaundice, vomiting, cyanosis, accelerated heart rate, shock and esthetic problems. Contamination of dyes also affects the photosynthesis process as the presence of color reduces light penetration (Bulut and Aydin, 2006). Hence the release of dyes waste into water should be monitored to control the dye concentration in the water system. Mostly, adsorption technique is used for the removal of dyes as it is more effective, highly significant, easy to handle and is able to remove the dyes in a wide concentration range (Kim et al., 2013).

The present work is focusing on the synthesis of modified carbon material and the removal of methylene blue (MB) from the aqueous media by using a sorption technique. Prior to analysis the wet oxidation method was applied to prepare iron impregnated carbon samples. For this purpose potassium permanganate ( $\text{KMnO}_4$ ) was used as the oxidizing agent. The oxidized surface was further treated with ferrous sulfate ( $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ ) solution. Both the AC and FeAC were characterized by using a number of characterization techniques. The adsorption studies covered the effects of initial concentration and temperature under different conditions.

## 2. Methods

### 2.1. Materials

All analytical grade materials were used without further purification. Activated carbon (AC) was purchased from R & M Chemicals, Essex (UK), while  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$  and sodium bicarbonate ( $\text{NaHCO}_3$ ) were provided by ACROS Organics, USA.  $\text{KMnO}_4$  was obtained from Amresco, USA and methylene blue ( $\text{C}_{16}\text{H}_{18}\text{ClN}_3$ ) was purchased from QReC, New Zealand.

### 2.2. Preparation of the iron impregnated activated carbon (FeAC)

Activated carbon was heated for 2 h at 110 °C before impregnation. A 5 g AC was stirred with 1 M  $\text{KMnO}_4$  solution, at a rate of 200 rpm for 20 min to allow for complete mixing. The suspension was then diluted with distilled water and filtered. The residue was reacted with ferrous sulfate solution (1 M  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ ) for 8 h and stirred at the same rate for thorough mixing. Afterwards, the suspension was filtered again, and washed with 1% sodium bicarbonate ( $\text{NaHCO}_3$ ) solution. The residue was then soaked in  $\text{NaHCO}_3$  solution overnight. The next day it was decanted and washed with distilled water. After air drying for 2 h it was kept in an oven for complete drying at 110 °C.

### 2.3. Characterization of AC and FeAC

The BET surface area and pore size distribution of both samples (AC and FeAC) were determined by measuring the nitrogen ( $\text{N}_2$ ) adsorption–desorption isotherms using surface area analyzer Model ASAP 2020 V4.01. To examine the elemental dispersion on the surface of both the samples, EDX analyses were conducted using Model LEO SUPRA 50VP INCAX. The  $\text{H}_2$ –TPR and  $\text{CO}_2$ –TPD profiles of both the samples (AC and FeAC) were obtained with Thermo-Finnigan TPD/R/O 1100 apparatus equipped with a thermal conductivity detector (TCD).

### 2.4. Sorption studies of methylene blue (MB) dye

Methylene blue sorption by AC and FeAC were analyzed at various initial concentrations (1–100 mg/L) and at different temperatures (298–328 K). A 0.1 g sample was taken and stirred with 40 mL aqueous solution of MB in a series of 100 mL flasks. The pH was adjusted by using  $\text{HNO}_3$  and  $\text{NaOH}$  (0.01–1 M). The

reaction mixtures were equilibrated for 4 h at 400 rpm using orbital shaker model IKA, KS 260 basic and magnetic stirrer WiseStir, DAIHAN Instrument Co. After equilibration the suspensions were filtered and the remaining MB concentration was determined by using Shimadzu-2600 UV–Visible spectrophotometer at  $\lambda_{\text{max}} = 660 \text{ nm}$ . The amount adsorbed (mg/g) and removal (%) of MB by AC and FeAC, were calculated by using the equations;

$$q_e = \frac{(C_o - C_e)V}{m} \quad (1)$$

$$\text{Removal (\%)} = \frac{(C_o - C_e)100}{C_o} \quad (2)$$

where  $C_o$  (mg/L) is the initial concentration,  $C_e$  (mg/L) is the concentration at equilibrium time  $t$  (min),  $V$  (L) is the methylene blue volume and  $m$  (g) is the weight of adsorbent used. All experiments were conducted in triplicates and average values are used.

## 3. Results and discussion

### 3.1. Characterizations of the adsorbents

The  $\text{N}_2$  adsorption–desorption isotherms were obtained to understand the porous structure of both the adsorbents. Both AC and FeAC, show small hysteresis loops in their isotherms at lower pressure referring to type II isotherms (Bakandritsos et al., 2004). The formation of hysteresis loop in the break through curves for  $\text{N}_2$  adsorption–desorption exhibit the meso-porosity in the material. The meso-pores distribution on the surface of sample FeAC can be attributed to the disordered stacking of carbon species. It further suggests the presence of both micro-pores (<2 nm) and meso-pores (2–50 nm) in the carbon matrices. The average micro-pore volumes ( $V_m$ ) of AC and FeAC were found to be 0.36 and 0.15  $\text{cm}^3/\text{g}$ , respectively. The lowering of micro-pore volume for FeAC is expected due to the conversion of micro-pore into the meso-pores due to the clogging of iron species into the pores on AC surface upon impregnation.

The BET surface area ( $S_{\text{BET}}$ ) of FeAC (543  $\text{m}^2/\text{g}$ ) is lower as compared to the AC (1094  $\text{m}^2/\text{g}$ ) after impregnation. However, the pore width of FeAC is larger (22.98 Å) as compared to the AC sample (17.23 Å) referring to the penetration of iron contents into the pores on the surface and thus occupying the available sites. The oxidation of carbon surface with different precursor material can result in an increase in the surface oxygen acidic groups and a decrease in the surface area. Lee and co-workers (2005) also reported iron doping into carbon materials resulted in a 22.5% reduction in the surface area of the modified AC. However, the observed values of the surface area for AC and FeAC (1094 and 543  $\text{m}^2/\text{g}$ , respectively) in the present study are much higher than reported by Atkinson et al. (2011) for porous materials prepared in a temperature range 500–600 °C (<40  $\text{m}^2/\text{g}$ ).

The EDX analysis confirmed the iron penetration into the pores and surface of AC sample and the increase of oxygen content after impregnation. The wt.% of C, O, Fe and S in FeAC sample were 48.68, 35.22, 13.75 and 1.14 wt.%, respectively. However, Mn and K were found in trace amounts i.e. 0.80 and 0.41 wt.%, respectively.

The  $\text{H}_2$ –TPR process illustrates the reduction (consumption of hydrogen as reducing gas) of the surface provided that there are active oxygen species on the substrate. The TPR profile of AC sample did not show significant reduction peak because of weak adsorption of  $\text{H}_2$  on the surface. In contrast, FeAC shows three reduction peaks at 484, 587 and 693 °C as the iron impregnation of AC has generated more oxygen groups on its surface, which is in agreement with the EDX analysis. The iron doping on AC results in the formation of different iron complexes e.g.,  $\text{Fe}_2\text{O}_3$ ,  $\text{Fe}_3\text{O}_4$ ,

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