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Effect of Al & Fe inorganic polymers on the enhancement of the surface properties of Activated carbon prepared from sesame stalks

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Abstract: Activated carbon (AC) has a high demand throughout the world that towards to minimize the cost of production and materials reuse. Recently, many researches have focused on the enhancement of the AC properties using various materials and methods. In this study, AC prepared from Sesame stalks (SSAC) and modified by using two inorganic polymers ($Al_x(OH)_y$ and $Fe_x(OH)_y$) to form AlAC and FeAC respectively. BET, SEM, EDX and FTIR were used to characterize the samples. The BET surface area of SSAC, AlAC and FeAC were 190.039, 206.167, 378.404 m^2g^{-1} respectively, and the volume of pores had increased in AlAC and FeAC comparing with SSAC. SEM showed that the surface of AlAC and FeAC were filled with some rod-like structures as it could be a result of the structural change of AC after Al and Fe polycations species adsorption. Overall, the analysis of the samples indicates that the inorganic polymers probably have a substantial effect on the structure and morphology of the samples, as well as indicate certain differences in their physical/chemical properties. The results prove that the modification of AC with inorganic polymers could be an interesting way to improve the AC properties and its adsorption capacity.

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1- Introduction

Activated carbon (AC) has a numerous applications long ago, such as catalysis, catalyst support (Diasa et al., 2007) or adsorbents for organic and inorganic contaminants and many other chemicals and organisms (Hung et al., 2005; Karanfil, 2006; Diasa et al., 2007; Gupta and Suhas, 2009; and Tran et al., 2017). The demand for AC is increasing through the world (Tran et al., 2017) due to its versatility, high adsorption capacity, low cost and its chemical/physical properties of surface which are the most important factors to define the characteristic of AC (Karanfil, 2006; Diasa et al., 2007; Cobb et al., 2012; and Jiang et al., 2015).

Presently, all world countries move forward to waste minimization and materials reuse, there were many research have been reported on the preparation of AC from agricultural wastes (Diasa et al., 2007), such as coconut shell (Cobb et al., 2012), sesame stalks (Jiang et al., 2015) and cotton stalks (Deng et al., 2009). Thus, agricultural wastes are considered favorable adsorbents for adsorption applications (Diasa et al., 2007; Gupta and Suhas, 2009; and Cobb et al., 2012).

As a global trend to improve the physical/chemical properties of adsorbents like AC, many techniques have been used through applying different methods such as impregnation, ozone, surfactant and microwave treatment (Uyak et al., 2007; Cho et al., 2011; Kristina et al., 2011; and Bhatnagar et al., 2013). Several studies showed that there were many attractive features of the modification of adsorbents such as AC and clays with various materials such as metal oxides, organic or inorganic polycations which could promote the potential of AC for specific contaminants depend on adapting surface properties after modification (Chen et al., 2007; Uyak et al., 2007; Cho et al., 2011; Kristina et al., 2011; and Nguemtchouin et al., 2015).

In addition, regarding the trivalent salts of aluminum and iron, that were the commonly used coagulants for several water and wastewater treatment operations, such as polyaluminum chloride (PACl), and polyferric chloride (PFC) (Jiang and Graham, 1998; and Shammas, 2005). These coagulants in the solutions were hydrolysis and give many polynuclear metallic hydroxides products contain high positive charge such as: for aluminum salt polymers are $Al_6(OH)_{15}^{3+}$ and $Al_7(OH)_{17}^{4+}$ and of iron salt polymers are $Fe_2(OH)_2^{4+}$ and $Fe_3(OH)_4^{5+}$, regarding polymerized hydroxyl complexes of aluminium it seems that $Al_{13}O_4(OH)_{24}^{7+}$ (often denoted by Al_{13} and found in solutions at pH 3.5-4.5) is the most effective and stable polymeric Al species in water treatment, these molecules are synthesized by titrating a relatively concentrated aluminum solution with a base at a hydrolysis ratio (OH^-/Al_{13}^+) that is maintained between 2.1-2.5 (Jiang and Graham, 1998; Shammas, 2005; and Abeysinghe, 2012).

These products contain high positive charge (Jiang and Graham, 1998; and Shammas, 2005), which creates good conditions for destabilisation of negatively charged contaminants which cause turbidity and color of water (Shammas, 2005; and Yan et al., 2008). Therefore, these can be considered as good candidates for enhanced anions removal besides heavy metals depending on the nature of AC surface. Moreover, it was found that organic cationic polymers could be used for modification of granular AC and enhancement of performances for anions removal from aqueous solutions (Cho et al., 2011). Thus, the aims of this study were: i) modification of AC, ii) assess the effect of this modification on surface morphology of AC. Consequently, this study is expected to help us better understand the role of the surface modification with inorganic polymers to the improved adsorption capacity of AC.

2. Materials and Methods

2.1. Preparation of Sesame Stalk Activated Carbon (SSAC)

Sesame stalk (SS) was collected from Sohag, Egypt. Sesame stalk (SS) was initially washed with distilled water to remove dust and subsequently dried at 110 °C for 24 h. The accurately weighted sample was carbonized at 300 °C in a muffle furnace (Fisher Scientific Isotempm Muffle Furnace) for 1.5 h. The SS charcoal product was crushed and sieved in order to get ≤ 2 mm size. Then, SS charcoal was mixed with NaOH within ratio of 1:2 (weight/weight), The charcoal was held in the solution for 24 h at room temperature and then dried at 110 °C for 24 h. The dried material was activated at 500 °C for 2 h in furnace at low oxygen atmosphere. The sample was left to cool down before taking out of the furnace. After activation, the sample obtained was washed with 0.1 M HCl, and then with distilled water to adjust the pH closer to 6.5 - 7. Then, SSAC sample was dried at 110 °C for 24 h and was stored in sealed flasks until used.

2.2. Preparation of Inorganic Polymers

Two inorganic polycations were obtained by addition of freshly prepared solutions of 0.1 M NaOH to 0.1 M aqueous solutions of $AlCl_3$ (Alpha Chemika) or 0.1 M $FeCl_3$ (Alpha Chemika), respectively. Then, the reactant was added drop wise at a quite slow addition rate (1 mL min^{-1}), until the reactant quantities reached to the molar ratios OH/Al = 2.4 and OH/Fe= 2 (Nguemtchouin et al., 2015). Rapid mixing (200 rpm) was necessary to prevent local over concentration of hydroxyl ions. Finally, the obtained solutions were aged for 2 days for aluminium and for 10 days for iron at room temperature (Nguemtchouin et al., 2015) before added to SSAC for modification.

2.3. Adsorption of Cationic Polymers on Activated Carbon

The following method has been done to modify SSAC with metallic polycations; Batch adsorption study was carried out at pH 2.3 by mixing 2.5 g/L SSAC with varying concentration of $Fe_x (OH)_y$ (where [Fe] was from 0.156 to 0.934 g/L) to reach equilibrium. The unadsorbed $Fe_x (OH)_y$ in the supernatant for each sample was evaluated by UV spectrophotometer at 340 nm (Lente, 2001).

The amount of $Fe_x (OH)_y$ adsorbed on the SSAC C_{ads} (mg/g) is calculated by following Eq.:

$$C_{ads} \quad \frac{(C_o \quad C_e)V}{W}$$

Where, C_o and C_e (mg/L) are the initial and equilibrium $Fe_x (OH)_y$ concentrations; W (g) is the adsorbent weight, and V (L) is volume of solution. The results were reported as the average of the three replicate experiments.

2.4. Preparation of Modified Activated Carbon (AlAC and FeAC)

The modification process of SSAC by $Fe_x (OH)_y$ was carried out under these conditions to prepare FeAC: 2.5 g of SSAC sample has pH 2.3, and concentration of $Fe_x (OH)_y$ (where [Fe] = 622 mgL^{-1}). As well to prepare AlAC, 2.5 g of SSAC sample has pH 4.4, and concentration of $Al_x (OH)_y$ (where [Al] = 622 mgL^{-1})). Then, the solution was stirring (150 rpm) for 5 h at room temperature. Finally the Products were separated by filtration paper and washed with distilled water until the washing solution was free of chloride ions (detected by $AgNO_3$ test). The products were dried at 303 K.

2.5. Materials Characterization

Some techniques such as BET, SEM, EDX and FTIR were used to evaluate SSAC, AlAC and FeAC samples. The surface morphological changes and elemental distribution at the surfaces of the samples were quantified by using a scanning electron microscope SEM and energy dispersive X-ray analyzer EDX (QANTA FEG 250). The BET surface area, total pore volume, and pore size distribution of samples were determined by N_2 at 77K using BET and

BJH methods (NOVA touch 4LX) (Brunauer et al., 1938; and Barrett et al., 1951). The FTIR spectra (4000-400 cm^{-1}) of the samples were performed using a (Nicolet IS 10) for characterization of functional groups on the surface of all samples.

3. Results and Discussion

3.1. Adsorption Isotherm of Cationic Polymer on Activated Carbon

The adsorption isotherms of the cationic polymers adsorption on the SSAC activated carbon surface were investigated mathematically by using both Langmuir and Freundlich isotherm equations (Sundaram, 2008; and Ahn et al., 2009): for adsorption on the SSAC activated carbon surface.

$$\frac{1}{q_e} = \frac{1}{K_L C_e} + \frac{a_L}{K_L} \qquad \text{(Langmuir)}$$

$$\log q_e = \log K_f + \frac{\log C_e}{n} \qquad \text{(Freundlich)}$$

where q_e (mgg^{-1}) is the concentration of [Fe] ions from $Fe_x\ (OH)_y$ on the surface of AC, and both a_L and K_L are Langmuir adsorption constants. The term C_e (mgL^{-1}) is the concentration of [Fe] ions from $Fe_x\ (OH)_y$ in the equilibrium, and both k_f and $(1/n)$ are Freundlich adsorption constants.

The relationship between the amounts of cationic polymer adsorbed (C_{ads}) and metal concentrations (C_e) at equilibrium was described by Langmuir (which proposes homogeneous monolayer adsorption on the surface of the adsorbent with a finite number of identified sites) and Freundlich (which based on adsorption onto a heterogeneous surface) isotherms models as shown in Table 1.

Table 1. Comparisons between Freundlich and Langmuir adsorption isotherm constants for $Fe_x\ (OH)_y$ onto

SSAC Freundlich adsorption isotherm constants		
K_f	n	R^2
0.000642	0.603136	0.9793
Langmuir adsorption isotherm constants		
K_L	a_L	R^2
0.000126	1.39876	0.9439

The result in Table 1 showed that Freundlich isotherm better than Langmuir isotherm equation in $Fe_x\ (OH)_y$ adsorption on SSAC, based on the coefficient of correlation (R^2) values.

The same concentration of ([Fe] = 622 mgL^{-1}) of Fex (OH)y and [Al] = 622 mgL^{-1}) of $Al_x\ (OH)_y$ during the modification step were added to SSAC to obtain FeAC and AlAC respectively and to easily compare the effect of different metallic polycations on surface of modified AC.

3.2. Surface Morphology Characterization

SEM images (Figure 1) showed that the surface of SSAC was filled with cracks, channels and some grains in the various porous sizes. It can be concluded from SEM images taken, that porous structure was formed because of most of the organic volatiles were evolved (Menendez-Diaz and Mart In-Gullon, 2006), leaving behind the ruptured surface of AC with a small number of pores during activation at 500 °C. Furthermore, the SEM micrographs of FeAC and AlAC has experienced a major change, which were filled with some rod-like structures that almost compactly dispersed as a result of the adsorption of Al and Fe polycations species on AC.

The EDX result provide information about the relative amounts of elements C, O, Al and Fe of the AC samples, all three samples contained C and O as major components; where the Atomic % of C were 89.21, 61.31 and 84.59, while the O content were 10.79, 35.49 and 14.28 for SSAC, AlAC and FeAC, respectively. As well the atomic % of Al and Fe were 3.2 and 1.14 for AlAC and FeAC respectively. Moreover, it was clear that the O ratio of SSAC modified samples increased with the increasing of O content of Al and Fe polycations species. Furthermore, the O content in AlAC higher than in FeAC and SSAC (approx twice). While the O content in FeAC was slightly higher than in SSAC.

3.3. BET Surface Characterization

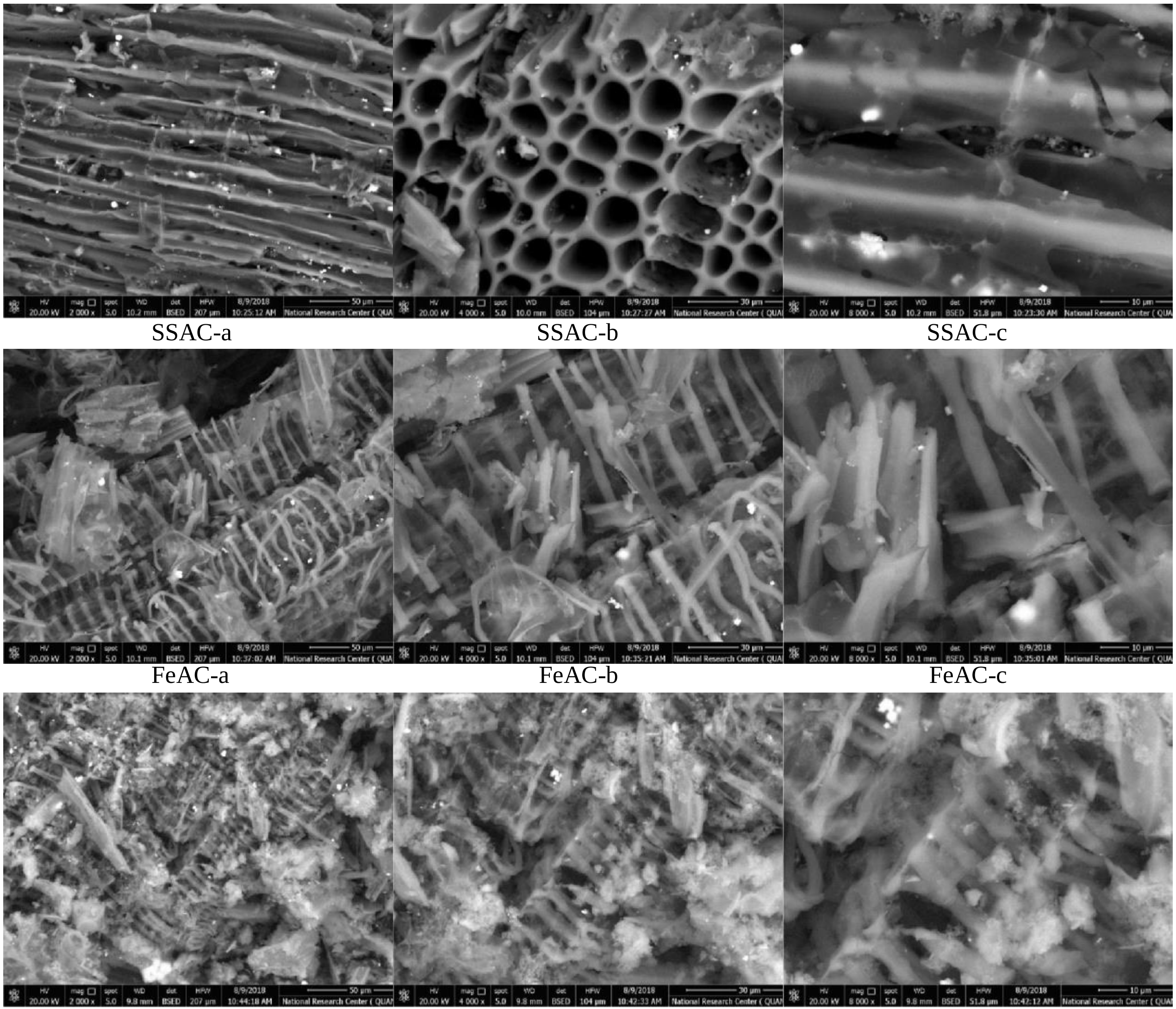
BET surface area results of SSAC, AlAC and FeAC were listed in Table 2. Clearly, the results obvious that there were increased in textural parameters of SSAC after modification by Al and Fe polycations.

According to the classification of IUPAC, the pore dimensions were classified into three groups: macropore (r > 50 nm), mesopore (r = 2–50 nm) and micropore (r < 2 nm). Thence, the pore radius was found to be about 1.9 nm with varied slightly of the three samples. Therefore, the AC sample and its modification were classified as micropores. Furthermore, the volume of pores was increased in AlAC and FeAC comparing to SSAC, which were substantial parameter conditional upon the type of both the medium and the pollutant that removed from it (Karanfil, 2006; and Diasa et al., 2007).

It is clear that the adsorption of the polycations species has a significant effect on the surface of SSAC (Table 2), where the specific surface area of SSAC, AlAC and FeAC were 190.039, 206.167 and 378.404

m^2g^{-1}, respectively. Moreover, the pore volumes of FeAC and AlAC are larger than that of SSAC.

The BET surface area of the SSAC relatively low (190.039 m^2g^{-1}). The result could be considered to be a good accordance due to an increasing burn off while activation in low oxygen atmosphere. Furthermore, slightly increase in the surface area of AlAC may be due to the formation of some precipitate in the solution of Al polycations (Jiang and Graham, 1998), led to relatively high reduced in the BET surface area compared to FeAC sample.


SSAC-a SSAC-b SSAC-c

FeAC-a FeAC-b FeAC-c

AlAC-a AlAC-b AlAC-c

Figure 1. Scanning electron micrographs (SEM) of SSAC, FeAC and AlAC in different an enlargement factor (a-2000x, b-4000x and c-8000x).

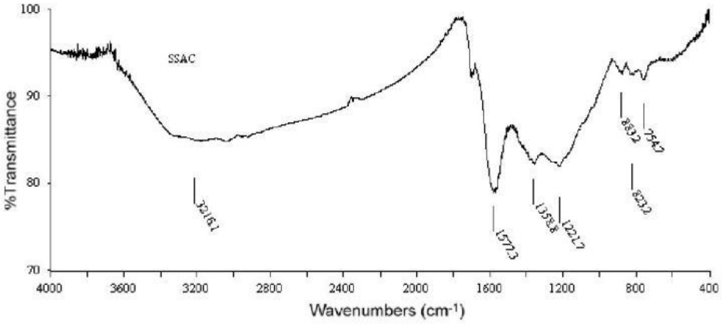
Table 2. BET surface area characterization of the SSAC, AlAC and FeAC samples.

Sample	Specific surface area (m^2g^{-1})	Total pore volume (cm^3 g^{-1})	Average Pore size (nm)	BJH Pore Size Distribution		
				pore area (m^2g^{-1})	Pore volume (cm^3g^{-1})	Pore radius (nm)
SSAC	190.039	0.12991	1.3672	23.6619	0.0410247	1.93644
AlAC	206.167	0.15488	1.5025	34.865	0.0617299	1.92216
FeAC	378.404	0.31572	1.6687	87.6158	0.151658	1.93023

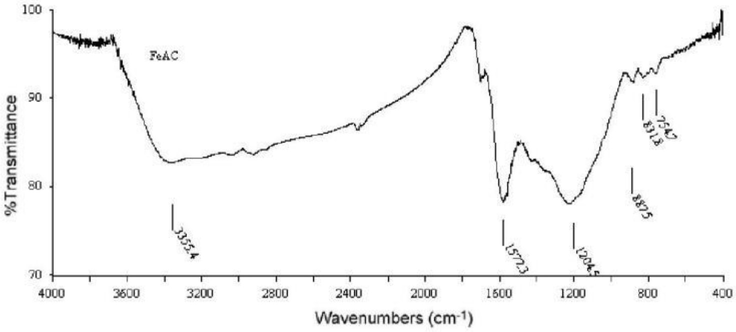
Both FeAC and AlAC textural parameters became more than SSAC that can be refer to the significant influence of the adsorption of Al and Fe polycations species on SSAC, which led to increase the adsorption capacities. Generally, whenever the adsorbent specific surface area became greater, it will enhance the adsorbent performance (Smisek and Cerny, 1970).

3.3. Infrared Spectroscopy

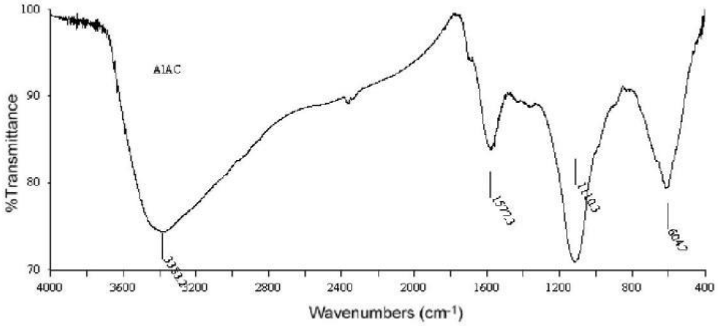
The possible chemical bonds in SSAC were investigated by the FT-IR spectra and were compared with FeAC and AlAC (Figure 2), the three samples showed almost similar FTIR spectra. Most of these bands have been reported by other investigators, all spectra exhibited a broad absorption peak in the range of 3200–3650 cm $^{-1}$ (3216 cm^{-1} for SSAC, 3355 cm^{-1} for FeAC and 3383 cm^{-1} for AlAC), which could be attributed to the stretching vibrations of –OH groups (Deng et al., 2009; and Sun et al., 2011).



SSAC



FeAC



AlAC

Figure. 2. FTIR spectra for SSAC, FeAC and AlAC.

The peak appearing at 1577 cm^{-1} for SSAC sample could be attributed to C=O stretching or to the bending vibration of water absorbed, polymerized and crystallized in the modified AC (Deng et al., 2009; and Sun et al., 2010; and Sun et al., 2011), also there were peaks at around 1350 and 1220 cm^{-1} may be due to oxygen functionalities such as highly conjugated C–O stretching (Deng et al., 2009).

Furthermore, it can be seen a weak bands at about 880, 820 and 760 cm^{-1} may be assigned to out of plane deformation vibrations of C-H group in aromatic structures (Rao et al., 2011). Moreover, the peaks at around 1200 cm^{-1} for FeAC could be attributed to the asymmetric stretching vibration of Fe–O–Fe (Sun et al., 2010).

The peak at 1110 cm^{-1} for AlAC and the peak at 831cm^{-1} for FeAC which were attributed to the asymmetric stretching vibration and bending vibration of Al–OH–Al or Fe–OH–Fe; also, there was a peak at 754 cm^{-1} for FeAC and a peak at 604 cm^{-1} for AlAC, which were attributed to bending vibrations of Fe–OH and Al–OH, respectively; indicating that the polymer are formed in the samples (Sun et al., 2010; and Sun et al., 2011). This probably indicates that the surface of AC could be positively charged which favors the adsorption of anions besides heavy metals depending on the nature of SSAC surface.

4. Conclusions

In the present study, AC from sesame stalks has been successfully synthesized followed by modifications using Al and Fe metallic polycations, These samples were characterized by SEM, EDX, BET and FT-IR analysis.

The SEM results showed that the surface of SSAC, AlAC and FeAC have a cracks, crevices, and some grains in various porous sizes which filled with some rod-like structures due to the formed metallic polycations species on the surface of SSAC.

The BET data showed that metallic polycations adsorption has a significant effect on the increasing of the specific surface area. Where the surface area of FeAC that has (378.404 $m^2.g^{-1}$) that about twice that of SSAC and AlAC. Moreover, the modification of SSAC enhances its adsorption capacity.

Furthermore, from FT-IR spectra, the absorption bands detected of the surface chemical groups like Al–OH–Al and Fe–OH–Fe that refer to the existence of polymerization and the complete surface modification process. Generally, These changes of surface properties indicate successful modification of activated carbon by the cationic polymers. Finally, it can be said that activated carbons prepared from sesame stalks and modification with metallic polycations could be used in the adsorption applications.

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