Mg-Modified Sugarcane Bagasse Biochar for Dual Removal of Ammonium and Phosphate Ions from Aqueous Solutions
Maher E. Saleh1 and Ramzy M. R. Hedia1

ABSTRACT
Utilization of agricultural wastes to generate cheap and efficient sorbents to remove contaminants from wastewaters is an up-to-date environmental challenge. In Egypt, sugarcane bagasse is yearly generated as a waste material in huge amounts. The objectives of this study were to investigate the effect of chemical modification on the properties of bagasse biochar generated from local sugarcane bagasse feedstock (SCBF) and assess its efficiency for removal of both ammonium and phosphate ions from artificial aqueous solutions. SCBB and Mg-SCBB biochars were produced through pyrolysis of raw SCBF and MgSO4 impregnated SCBF, respectively at 500 °C and under oxygen-limited condition. FTIR peaks analysis, DEM examination, and some physical and chemical properties revealed that new surface functional groups, meso- and micropores, larger surface area and higher CEC were developed in SCBB and Mg-SCBB compared to SCBF. SCBB and Mg-SCBB showed high affinity to ammonium adsorption from aqueous solutions comparable to Charcoal and Zeolite. Mg-SCBB was the only sorbent capable of removing phosphate from the aqueous. Ammonium and phosphate removal at 1:200 sorbent to solution ratio were higher than those at 1:500 for all sorbents. A slight ammonium volatilization occurred during the adsorption process due to high solution pH. Adsorption kinetics data were best fitted to the pseudo-second-order kinetic equation suggesting intraparticle diffusion controlled adsorption process. Ammonium adsorption isotherms were best fitted to Freundlich model. The calculated Freundlich intensity parameter (n) ranged from 0.478 to 0.894 indicating favorable adsorption of ammonium and phosphate by all sorbents. Mg-SCBB had an adsorption capacity of 2573.9 and 4002.2 mg kg⁻¹ for ammonium and phosphate, respectively. The produced Mg-modified sugarcane bagasse biochar may represent a promising efficient and cheap sorbent for dual remediation of wastewaters contaminated with ammonium and phosphate ions.

Keywords: Sugarcane bagasse, Mg-modified biochar, ammonium and phosphate removal, agricultural wastes, aqueous solutions.

INTRODUCTION
Due to fast industrialization and the widespread application of agrochemicals, the occurrence of organic and inorganic pollutants has considerably increased in our environment and food chain. Therefore, serious public and governmental concerns about the environmental pollution and human health have been raised (WHO, 2017). Due to inefficient management of agricultural fertilizers and large discharge of wastewaters, the concentrations of various nutrients (NH₄⁺, NO₃⁻ and PO₄³⁻) are largely increased in surface and groundwater resources globally (Carey et al., 2015). Freshwater containing elevated nutrients concentrations discharges into oceans depleting dissolved oxygen concentrations and promoting harmful algal blooms. This creates hypoxic ‘dead zones’ and causes ecological damage (Dunnivant and Anders, 2006).

Ammonium ion has a positive charge and a much lower charge-to-radius ratio than many metal cations. Therefore, NH₄⁺ is mainly attracted to the negatively charged surfaces of colloidal particles and behaves similarly to alkal metal cations (e.g. Na⁺, K⁺). Unlike divalent metal cations, NH₄⁺ does not form stable bonds with surfaces to which it is attached. However, the oxyanions NO₃⁻ and PO₄³⁻ are negatively charged and thus attracted to positively charged surfaces. With different behavior to each other, NO₃⁻ is much more mobile while PO₄³⁻ is readily bound to surfaces of colloidal particles in water (Conley et al., 2009). Common methods of remediation polluted aqueous phases include ion exchange, precipitation, membrane separation and adsorption (using activated carbon) techniques among others. These techniques are expensive and produce chemical residues which have uneconomic value.

Biochar is a carbonaceous, fine-grained, porous solid material produced by pyrolysis of a wide variety of biomasses under different conditions. As a low-cost substitute of activated carbon, biochar can be used as carbon sequestration and soil amendment (Carey et al., 2015; Agegnehu et al., 2017). Besides, biochar is recently used as a green environmental sorbent to remove diverse organic contaminants such as aromatic dyes, antibiotics, polychlorinated biphenyls (PCBs), polycyclic aromatic hydrocarbons (PAHs), volatile organic compounds (VOCs) and agrochemicals from aqueous and gaseous phases (Qiu et al., 2009; Beesley et al., 2010; Zheng et al., 2010; Teixido et al., 2011; Xu et al., 2011; Saleh et al., 2016; De Jesus et al., 2017), or inorganic contaminants such as heavy metals and various nutritional elements (NH₄⁺, NO₃⁻ and PO₄³⁻) from urban and industrial wastewaters and agricultural drainage water (Fang et al., 2014; Saleh et al., 2012;
Ding et al. (2014) found that SCBB produced at different pyrolysis temperatures (from 250 to 600 °C) was effective in Pb\textsuperscript{2+} sorption from aqueous solutions and the sorption capacity of the produced biochar decreased with increasing the temperature of pyrolysis (from 2.1 to 6.1 mg Pb/g biochar at 250 and 600 °C, respectively). The initial, rapid Pb sorption was probably controlled by ion exchange and/or complexation then it was slowed down due to intraparticle diffusion. Noraini et al., (2016) reported that magnetic biochar produced from sugarcane bagasse by microwave heating at optimum conditions of 30 min of radiation time and Fe\textsubscript{2}O\textsubscript{3} impregnation ratio 0.45 (Fe\textsubscript{2}O\textsubscript{3} : biomass), could enhance the removal efficiency of Cd\textsuperscript{2+} to 96.17% from aqueous solutions. Carrier et al. (2012) obtained a high-quality SCBB was also produced by vacuum pyrolysis and steam activation having an SSA of 418 m\textsuperscript{2} g\textsuperscript{-1} and a cation exchange capacity (CEC) of 122 cmol kg\textsuperscript{-1}. This biochar was very efficient in methylene blue (MB) adsorption and favored physisorption rather than chemisorption.

The efficiency of nitrate adsorption by SCBB, generated at 300 °C for 4 h in a carbonization furnace, was enhanced by chemical modification of air-dried bagasse using epichlorohydrin, dimethylformamide and ethylenediamine combination before pyrolysis (Hafshejani et al., 2016). Adsorption capacity for nitrate removal from aqueous solutions was increased from 11.56 to 28.21 mg g\textsuperscript{-1} for SCBB and modified SCBB, respectively. The maximum capacity (q\textsubscript{max}) of nitrate was reached with sorbent dose of 2 g L\textsuperscript{-1}, equilibrium pH 4.64 and after 60 min of contact time. Schwantes et al. (2015) showed that SCBB produced from 3 M H\textsubscript{3}PO\textsubscript{4}-activated bagasse was efficient in NO\textsubscript{3}\textsuperscript{-1} removal from aqueous solutions. The adsorption isotherms followed the Freundlich model and sorption kinetics data were fitted to the pseudo-second-order equation.

Trazzi et al. (2016) found that surface area and fixed carbon content of SCBB were increased with increasing pyrolysis temperature and residence time. The best performance was observed for SCBB formed at 500 °C for 60 min. in terms of energy expended and phosphate sorption. Adsorption on of phosphate was found to be non-spontaneous, endothermic process and adsorption capacity increased with temperature.

Therefore, the objectives of this study were to investigate the effect of chemical modification on the properties of bagasse biochar generated from local sugarcane bagasse and to assess its efficiency for removal of both ammonium and phosphate ions from aqueous solutions.
MATERIALS AND METHODS

Preparation of feedstock:

Sugarcane bagasse feedstock (SCBF) was collected from the local market in Alexandria Governorate. SCBF was cut into 2.0 cm segments, washed with tap water, then with distilled water and oven-dried at 70 °C for 24 h. The oven-dried SCBF was left to cool to reach the ambient lab temperature and then stored in a plastic vessel for the pyrolysis step. A slight modification to the method outlined by Fang et al. (2014) was made to prepare Mg-saturated SCBF: A portion of the oven-dried SCBF was soaked in 1.6 M MgSO₄·7H₂O at 1:10 solid to solution ratio for 2 h. The Mg-saturated SCBF (Mg-SCBF) was then oven-dried at 80 °C for 48 h, left to cool to ambient temperature and then stored in a plastic vessel for the pyrolysis step.

Generation of biochar:

Biochar was generated by heating SCBF and Mg-SCBF under oxygen-limited condition (pyrolysis). For pyrolysis, 200 g of both previously prepared SCBF and Mg-SCBF was tightly wrapped in an aluminum foil sheet. The wrappings were perforated with five tiny holes to allow pyrolytic gases to escape and facilitate an oxygen-limited condition. Pyrolysis was conducted in a muffle furnace by increasing temperature at a rate of 19 °C/min to reach 500 °C and then heating was continued at 500 °C for 60 min. The generated bagasse (SCBB) and the Mg-modified bagasse biochars (Mg-SCBB) were left to cool to room temperature and the produced carbonaceous material was weighed. Afterwards, biochars were ground in a porcelain mortar and pestle and sieved through 0.5 mm polyethylene sieve. The biochars were then washed several times with distilled water, oven-dried at 80 °C for 24 h, left to cool to room temperature and finally stored in plastic vessels for subsequent experiments.

Characterization of sorbents:

Percent of biochar yield and volatile matter were calculated from the oven-dried SCBF and the generated carbonized materials after pyrolysis. The pH was measured (pH-meter, Inolab pH/Ion 735) in the suspensions of 1.0 g biochar in 100 ml of distilled water. The electrical conductivity (EC, dS m⁻¹) was measured using a conductivity meter (WTW inoLab Cond 720, GmbH) in the filtrate of a 5% W/V solid/distilled water suspension. Surface area (m² g⁻¹) of the biochars was determined using the methylene blue method (Kaewprasit et al., 1998). The ash content of biochars was determined according to Samsuri et al. (2014) in open porcelain crucibles using the muffle furnace at 700 °C for 12 hrs. Cation exchange capacity (CEC) was determined using the modified ammonium acetate compulsory displacement procedure (Gaskin et al., 2008). The CHNS Elemental Analyzer (Vario, type EL) was utilized to determine the carbon, hydrogen, nitrogen, and sulfur contents.

Fourier transform infrared (FTIR) spectra of the biochars and bagasse feedstock were established in the range 400–4000 cm⁻¹ (SHIMADZU infrared spectrophotometer; model FT/IR-5300, JASCO Corporation) to determine predominant surface functional groups. Images of a scanning electron microscope (SEM, Jeol model 6360 OLA) was used to investigate the surface micro-morphology of biochars.

For comparison, Zeolite and Charcoal, as commercial adsorbents, were purchased from private companies to be used for the comparison with the generated biochars for their efficiencies of ammonium and phosphate ions removal from aqueous solutions.

Stock solution of ammonium and phosphate:

A stock solution containing 500 mg P/l and 580 mg NH₄⁺/l was prepared using an analytical grade of di-ammonium orthophosphate salt (2.129 g (NH₄)₂HPO₄/l). From the stock solution, aqueous solutions with a series of NH₄⁺ and P concentrations were prepared for the proposed adsorption experiments.

Kinetics of ammonium and phosphate sorption:

The kinetics of ammonium and phosphate removal by the four sorbents (SCBB, Mg-SCBB, Zeolite, and Charcoal) from aqueous solutions was investigated using the compartment method. Artificial aqueous solutions containing 23.2 ppm NH₄⁺ and 20 ppm P (representing average concentrations of such species in local municipal wastewater) were prepared from the stock solution. In a double jacket reaction vessel (Fig. 1), 2 or 5 g of each sorbent was suspended in 1000 ml aqueous solution to represent 1:200 or 1:500 sorbent to solution ratio. The reaction took place in the inner vessel. The suspension was continuously stirred at 500 rpm using magnetic stirrer and temperature of the suspension was kept constant at 25°C using a continuous circulating thermostatic water system running in the outer vessel and surrounding the reaction inner vessel. Subsamples of suspension (20 ml) were withdrawn from the reaction vessel using a manual syringe at time intervals of 1, 2, 3, 4, 6, 8, 10, 12 and 24 h and pH of the sampled suspensions was immediately measured. The suspensions were then filtered through a filter paper (Whatman no. 1) and the equilibrium concentrations of ammonium and phosphate were measured in the filtrates. The concentrations of ammonium and phosphate were determined colorimetrically according to the methods described by Keeney and Nelson (1982) and Murphy and Riley (1962), respectively.
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Fig. 1. The double-jacket reaction vessel used in the adsorption experiments

The amount of ammonium and phosphate adsorbed per sorbent unit mass \((q, \text{ mg g}^{-1})\) were calculated using equation (1).

\[
q = \frac{(C_0 - C_{eq})}{m} v
\]  
(1)

Where \(C_0\) and \(C_{eq}\) are the initial and equilibrium concentrations (mg/l), respectively, \(m\) is the mass of sorbent material (g), and \(v\) is the volume of aqueous solution (l).

For correction of ammonia volatilization, the evolved ammonia from the reaction vessel at each time interval was collected and trapped in 25 ml of boric acid for its measurement. Plots of sorption kinetics (time vs. \(q\)) were established and the equilibrium time was evaluated. The fitness of these plots to the first- and second-order mathematical models of reaction kinetics was also investigated.

The removal percentage of adsorbate \((R, \%)\) was also calculated from Equation (2) as follows:

\[
R\% = \frac{(C_0 - C_{eq})}{C_0} \times 100
\]  
(2)

Isotherms of ammonium and phosphate sorption:

Sorption isotherms of \(\text{NH}_4^+\) and \(\text{P}\) on the investigated sorbents were established using the 1:200 and 1:500 suspension ratios and a series of initial concentrations of \(\text{NH}_4^+\) (23.2, 46.4 and 92.6 ppm) and \(\text{P}\) (20, 40 and 80 ppm). The sorption reaction was conducted for each individual sorbent under the same conditions of the aforementioned kinetic experiment (stirring at 500 rpm and temperature at 25 °C).

Suspensions were sampled at the equilibrium time determined from the kinetic experiment. Similarly, pH was measured immediately in the suspension and \(\text{NH}_4^+\) and \(\text{P}\) were also measured in the filtrate using the same methods. Ammonia volatilization was also determined for the correction of sorbed \(\text{NH}_4^+\). Sorption isotherms were plotted \((q\text{ vs. } C_{eq})\) and the fitness to Freundlich and Langmuir adsorption mathematical models were tested.

Mathematical Models

Kinetic models:

The model expressed in Equation (3) was used to describe the intra-particle diffusion as the controlling step for the adsorption process (Weber and Morris, 1963; Zhang et al., 2015).

\[
q_t = K_p t^{1/2} + C
\]  
(3)

Where \(k_p\) is the rate constant of intra-particle diffusion (mg/g min\(^{1/2}\)), and \(C\) (mg/g) is the intercept. A linear plot of \(q_t\) against \(t^{1/2}\) can be used to test the goodness-of-fit of the experimental data to the pseudo-first-order model.

The Lagergren pseudo-first-order kinetic model can be expressed by Equation (4)

\[
\frac{dq_t}{dt} = k_1 (q_e - q_t)
\]  
(4)

Where \(k_1\) is the adsorption rate constant (g/min), \(q_e\) is the amount of phosphate adsorbed at equilibrium (mg/g), and \(q_t\) is the amount of adsorbed phosphate at time \(t\) (mg/g). For limits of the initial conditions from
q<sub>e</sub> = 0, at t = 0 to q<sub>e</sub> at time t, the integration of Equation (5) yields:

\[ q_t = q_e \left[ 1 - \exp(-k_1 t) \right] \quad (5) \]

The linearized form of this integral can be expressed in Equation (6):

\[ \ln(q_t - q_e) = \ln q_e + k_1 t \quad (6) \]

Where \( q_e \) and \( k_1 \) can be obtained from the regression plot of \( \ln(q_t - q_e) \) against t.

The pseudo-second-order model (Ho and McKay, 1999; Ho, 2006) can be expressed by Equation (7):

\[ \frac{dq_t}{dt} = k_2 (q_e - q_t)^2 \quad (7) \]

The integration of the of Equation (7) for the initial conditions \( q_e = 0 \) at \( t = 0 \) yields

\[ q_t = \frac{q_e^2}{1 + q_e k_2 t} \quad (8) \]

Where \( k_2 \) is the adsorption rate constant (g/mg min), \( q_e \) is the amount of adsorbed phosphate (mg/g) at equilibrium, and \( q_t \) is the amount of adsorbed phosphate at time \( t \) (mg/g).

The linearization of Equation (8) was developed in different versions (Ho, 2006; Ghasemi et al., 2013; Plazinski et al., 2013) as presented in Table (1).

### Table 1. Different versions of pseudo-second-order model used for adsorption kinetics studies

<table>
<thead>
<tr>
<th>Linearized versions</th>
<th>Equation No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \frac{t}{q} = \frac{1}{k q_e^2} + \frac{1}{q_e} t )</td>
<td>8 – 1</td>
</tr>
<tr>
<td>( \frac{1}{q} = \frac{1}{q_e} + \left( \frac{1}{k q_e} \right) + \frac{1}{t} )</td>
<td>8 – 2</td>
</tr>
<tr>
<td>( q = q_e - \left( \frac{1}{k q_e} \right) \frac{q}{t} )</td>
<td>8 – 3</td>
</tr>
<tr>
<td>( \frac{q}{t} = k q_e^2 - k q_e q )</td>
<td>8 – 4</td>
</tr>
</tbody>
</table>

### Models for sorption isotherms:

The empirical Freundlich linearized model expressed in Equation (9) was used to describe the relation between the amount of adsorbed ammonium or phosphate (\( q_e \)) on biochar and their concentration (\( C_e \)) at equilibrium.

\[ \log q_e = \log k_f + \frac{1}{n} \log C_e \quad (9) \]

Where \( k_f \) and \( 1/n \) are the constants that reflect the capacity and intensity of adsorption. These constants can be calculated from the plots of \( \log q_e \) against \( \log C_e \), were \( 1/n \) equals the slope of the straight line and \( K_f \) equals its intercept.

Also, the commonly applied Langmuir linearized model is expressed in Equation (10).

\[ \frac{C_e}{q_e} = \frac{C_e}{q_{max}} + \frac{1}{q_{max} K_L} \quad (10) \]

Where \( q_{max} \) is the maximum monolayer adsorption capacity (mg kg<sup>-1</sup>) and \( K_L \) is a constant that reflects the favorability of the adsorption process. The plots of \( C_e/q_e \) against \( C_e \) give a straight line whose slope equals \( 1/q_{max} \) and the intercept equals \( 1/q_{max} K_L \).

Data of the sorption kinetics experiments were analyzed to find the most successful mathematical model to describe the removal of ammonium and phosphate by the generated sugarcane bagasse biochars. The least squared differences technique was applied and the Coefficient of determination (R<sup>2</sup>) was used as the statistical criteria for the goodness-of-fit of the tested models to the experimental data.

### RESULTS AND DISCUSSION

#### Characteristics of generated biochars:

**Physical characteristics:**

When 200 g of sugarcane bagasse feedstock (SCBF) was soaked in the MgSO<sub>4</sub> solution and then dried at 80°C, the final weight obtained was 302.82 g. The percent increase in the Mg-impregnated feedstock was 54.41% and it was due to the absorbed amount of MgSO<sub>4</sub> salt within the capillary tissue of the sugarcane bagasse. After pyrolysis at 500 °C, 51.16 and 105.93 g of SCBB and Mg-SCBB were obtained, respectively. Thus, the calculated biochar yields from feedstock were 25.58 and 34.98 % on the dried mass basis (Table 2). This was due to dehydration and condensation processes. Volatile matter contents were calculated to be 74.42 and 65.02 % for SCBB and Mg-SCBB, respectively. SCBB ash and moisture contents were lower than those of Mg-SCBB due to the higher Mg content of the latter. The generated SCBB biochar had a black color and Mg-SCBB had a dark gray color and higher ash content due also to its high Mg content.

**Chemical characteristics:**

The generated SCBB and Mg-SCBB had higher pH values than SCBF (Table 2) and Mg-SCBB was more alkaline than SCBB. In addition, the EC values (dS m<sup>-1</sup>) of SCBB and Mg-SCBB were slightly higher compared to its value for SCBF feedstock (Table 2). Carbon, hydrogen, nitrogen, and sulfur contents of biochars were higher than the bagasse feedstock due to condensation and they were comparable to those.
Table 2. Main physical and chemical characteristics of sugarcane bagasse feedstock and the tested sorbents

<table>
<thead>
<tr>
<th>Criteria</th>
<th>SCBF Feedstock</th>
<th>Biochars SCBB</th>
<th>Biochars Mg-SCBB</th>
<th>Charcoal</th>
<th>Zeolite</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Physical characteristics</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Biochar yield, %</td>
<td>-</td>
<td>25.58</td>
<td>34.98</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Volatile matter, %</td>
<td>-</td>
<td>74.42</td>
<td>65.02</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Ash, %</td>
<td>-</td>
<td>15.61</td>
<td>20.64</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Color</td>
<td>-</td>
<td>black</td>
<td>dark gray</td>
<td>black</td>
<td>white</td>
</tr>
<tr>
<td>Surface area, m² g⁻¹</td>
<td>-</td>
<td>38.74</td>
<td>42.12</td>
<td>35.61</td>
<td>49.87</td>
</tr>
<tr>
<td><strong>Chemical characteristics</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>EC, dS m⁻¹</td>
<td>0.70</td>
<td>0.95</td>
<td>1.02</td>
<td>0.87</td>
<td>0.74</td>
</tr>
<tr>
<td>pH</td>
<td>3.36</td>
<td>7.09</td>
<td>9.88</td>
<td>8.95</td>
<td>6.95</td>
</tr>
<tr>
<td>CEC, cmol kg⁻¹</td>
<td>-</td>
<td>34.74</td>
<td>39.57</td>
<td>31.24</td>
<td>48.02</td>
</tr>
<tr>
<td>C, %</td>
<td>42.82</td>
<td>67.61</td>
<td>64.35</td>
<td>65.84</td>
<td>-</td>
</tr>
<tr>
<td>N, %</td>
<td>0.55</td>
<td>0.79</td>
<td>0.73</td>
<td>0.76</td>
<td>-</td>
</tr>
<tr>
<td>C/N ratio</td>
<td>77.85</td>
<td>85.6</td>
<td>88.2</td>
<td>86.63</td>
<td>-</td>
</tr>
<tr>
<td>S, %</td>
<td>0.42</td>
<td>0.39</td>
<td>0.34</td>
<td>0.41</td>
<td>-</td>
</tr>
</tbody>
</table>

obtained by many researchers (Saleh et al., 2013; Jindo et al., 2014; El-Gamal et al., 2017). The elemental contents for Mg-

SCBB were slightly lower compared to SCBB presumably due to its higher Mg content. Condensation led also to the increase in the C/N ratio from 77.85 for bagasse feedstock to 85.6 and 88.2 for SCBB and Mg-

SCBB, respectively. Sulfur content was slightly reduced after pyrolysis (Table 2).

**Surface functional groups by FTIR analysis:**

The FTIR spectra of SCB feedstock and the generated biochars SCBB and Mg-SCBB are presented in Fig. (2). Generally, the peaks obtained for the three materials are different in their wave numbers peaks and intensities. Peaks of O-H stretching of free hydroxyl group were observed within the range from 3815.4 and 3786.39 cm⁻¹, from 3875.12 to 3757.46 cm⁻¹, and from 3861.62 to 3734.31 cm⁻¹ for SCBF, SCBB, and Mg-

SCBB, respectively indicating the chemically adsorbed water molecules and surface hydroxyl groups, SCBF had the highest intensity of free OH peaks and it was much lesser in SCBB and Mg-SCBB (Saleh et al., 2013). This may indicate the loss of free water molecules from SCBF during the pyrolysis process at 500 °C.

The presence of OH groups and H-bonds bands of alcohols and phenols were observed at peaks 3410.26, 3446.91, and 3396.76 cm⁻¹ for SCBF, SCBB, and Mg-

SCBB, respectively (Jindo et al., 2014). The intensities of these peaks were the highest for Mg-SCBB (Daffalla et al., 2010). This may be due to the initial impregnation of SCBF with a MgSO₄ solution, heating at 105 and the formation of MgO due to SO₂ gas evolution and the formation of a surface precipitate of metal oxide and hydroxide surfaces (Claoston et al., 2014; Sizmur et al., 2017).

Peaks assigned to aliphatic C-H vibrational stretching appeared in SCBF spectrum at 2921.61 cm⁻¹, indicating the presence of alkanes. However, this peak was not detected in SCBB or in Mg-SCBB spectra. This may indicate dehydration of cellulosic and ligneous components and thus the dehydrogenation of methylene groups of bagasse feedstock which led to a condensed structure of the generated biochars. Peaks of stretching C=C existed in SCBF, SCBB, and Mg-SCBB spectra at 1633.76, 1583.61, and 1660.77 cm⁻¹ assigned to C=O groups stretching were detected in SCBF, SCBB, and Mg-

SCBB (Harvey et al., 2012; Saleh et al., 2013 and Yao et al., 2013).

Aromatic C=C stretching bands, an indication of benzene-like rings, were also indicated by the peaks at 1402.30, 1417.73, and 1446.66 cm⁻¹ in SCBF, SCBB, and Mg-SCBB spectra, respectively, with higher peak intensity in SCBB and Mg-SCBB spectra (Harvey et al., 2012; Saleh et al., 2013; El-Gamal et al., 2017). Bands appeared at 500-700 cm⁻¹ may indicate the presence of halide-alkyl vibrational stretching and/or inorganic metal compounds such as of Ca, Mg, or K (Claoston et al., 2014).

Similarities and differences in the characteristic surface functional groups of SCBF and the generated biochars reveal that changes occurred in the molecular structure and surface configuration of the generated biochars leading to the loss of aliphatic, alcohol and...
Fig. 2. FTIR spectra of SCB feedstock (top), SCBB (middle), and Mg-SCBB (bottom)
alkane compound, sugar, and cellulosic and the increase in the aromaticity as temperature increases during pyrolysis (Liu et al., 2015; Carrier et al., 2012; Saleh et al., 2013; El-Gamal et al., 2017).

**Microstructure using SEM imaging.**

The microstructure of SCBF, SCBB, and Mg-SCBB was investigated by SEM imaging (Fig. 3). It can be observed that SCBF has a complex porous network and that it has fibrous, smooth surfaces in which macropores are dominant. However, pyrolysis at 500 °C led to coarse surfaces of the generated biochars, especially Mg-SCBB and a more porous structure can be seen in the SEM images. More meso- and micropores in the generated SCBB biochar were developed on the account of macropores. Micropores were even increased in Mg-SCBB biochar compared to SCBB. The development of these micropores in Mg-SCBB may explain the higher surface area measured for this modified biochar (Table 2). Due to MgSO₄ impregnation, surface coverage with Mg(OH)₂ or MgO crystals could also be identified on
Mg-SCBB surfaces compared to SCBB. Changes in the pore configurations of the generated biochars compared with the feedstock are presumably due to the thermal decomposition of cellulose and hemicellulose during the pyrolysis process leaving the cell walls which has higher proportions of lignin compounds. These results are similar to those obtained by Bonelli et al. (2006), Carrier et al. (2012), Harvey et al. (2012), Saleh et al. (2013); Liu et al. (2015), and El-Gamal et al. (2017) who observed similar changes in the surface morphology and size distribution of pores of biochars generated by the pyrolysis of sugarcane bagasse and other agricultural waste materials. These changes were found to be dependent on the type of feedstock, pyrolysis temperature, and the pre- or post-pyrolysis treatments (El-Gamal et al., 2017; Sizmur et al., 2017).

Adsorption kinetics of ammonium and phosphate ions:

Results of adsorption kinetics of ammonium ions (Fig. 4) revealed that sorption of ammonium from aqueous solutions by the tested sorbents was very fast during the first two hours. SCBB and Mg-SCBB were capable of removing ammonium from the aqueous solution at both 1:200 and 1:500 sorbent to solution ratios. For 1:200 ratio and 23 mg/l of initial ammonium concentration, the maximum ammonium removal percent had the order: Zeolite > Mg-SCBB > SCBB > Charcoal (96.41, 80.38, 67.87, and 61.81%, respectively) and were achieved after 12 h of contact time. However, the ammonium removal percents for these sorbents were very close to each other at 1:500 ratio and they were generally lower than those at 1:200 (72.78, 52.58, 51.13, and 50.19%, for Zeolite, Mg-SCBB, SCBB, and Charcoal, respectively). The developed meso- and micropores in the porous structure, due to modification by MgSO₄ impregnation of SCBF, enhanced the efficiency of Mg-SCBB to remove ammonium from solution compared with SCBB and it performed comparably to Zeolite and Charcoal. Similar results were obtained Yao et al. (2013), Fang et al. (2014) and Takaya et al. (2016) who found that pretreatments of biomasses with metal solutions enhanced the efficiency of the generated biochars in removing inorganic ions (NH₄⁺, NO₃⁻, PO₄-P) from aqueous solutions.

Ammonia volatilization continued during the ammonium adsorption process due to the alkaline conditions of the solution (Fig. 5). Mg-SCBB exhibited the highest cumulative ammonia volatilization. This could be explained by its strong alkaline effect due to MgO hydrolysis compared with the other sorbents (Fig. 6). However, SCBB and Zeolite recorded the least cumulative ammonia volatilization due to their lower solution pH. Cumulative ammonia volatilization for the tested sorbents did not change considerably between the 1:200 and 1:500 ratios.

---

Fig. 4. Percent of NH₄⁺ removal from aqueous solutions by different sorbents at A) 1:200, and B) 1:500 sorbent: solution ratios
Fig. 5. Cumulative percent of ammonia volatilization during NH$_4^+$ sorption from aqueous solutions by different sorbents at A) 1:200, and B) 1:500 sorbent: solution ratios.

Fig. 6. Changes in pH during NH$_4^+$ or P removal from aqueous solutions by different sorbents at A) 1:200, and B) 1:500 sorbent: solution ratios.
and removal was higher at reached after percent of phosphate by Mg or SCBB, it was only evident for Mg solutions was not observed at all for Zeolite, Charcoal
89.59 order: Zeolite > Mg 12 volatilized) was calculated for the tested sorbents after
Table 3. Regression parameters of fitting NH₄⁺ and P adsorption kinetics data to different linear forms of pseudo-second-order equation

<table>
<thead>
<tr>
<th>Sorbate/Sorbent</th>
<th>Ratio</th>
<th>Eq.</th>
<th>Slope</th>
<th>Intercept</th>
<th>R²</th>
<th>Sorbate/Sorbent</th>
<th>Ratio</th>
<th>Slope</th>
<th>Intercept</th>
<th>R²</th>
</tr>
</thead>
<tbody>
<tr>
<td>NH₄/SCBB</td>
<td>1:500</td>
<td>6-1</td>
<td>1.0E-04</td>
<td>1.0E-04</td>
<td>0.997</td>
<td>P/Mg-SCBB</td>
<td>1:200</td>
<td>2.0E-04</td>
<td>2.0E-04</td>
<td>0.991</td>
</tr>
<tr>
<td></td>
<td></td>
<td>6-2</td>
<td>5.0E-05</td>
<td>1.0E-04</td>
<td>0.628</td>
<td></td>
<td></td>
<td>4.0E-04</td>
<td>2.0E-04</td>
<td>0.901</td>
</tr>
<tr>
<td></td>
<td></td>
<td>6-3</td>
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<td>7419.6</td>
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<td></td>
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<td></td>
<td>6-4</td>
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<td>-0.7464</td>
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<td>NH₄/SCBB</td>
<td>1:200</td>
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<td>3.0E-04</td>
<td>1.0E-04</td>
<td>0.999</td>
<td>NH₄/Zeolite</td>
<td>1:500</td>
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<td>3.0E-05</td>
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<td>1.0E-04</td>
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<td>0.914</td>
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<td>NH₄/Mg-SCBB</td>
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<td>1.0E-04</td>
<td>4.0E-05</td>
<td>1.000</td>
<td>NH₄/Zeolite</td>
<td>1:200</td>
<td>2.0E-04</td>
<td>1.0E-04</td>
<td>0.998</td>
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<td>0.802</td>
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<td>1:200</td>
<td>1</td>
<td>3.0E-04</td>
<td>1.0E-04</td>
<td>0.999</td>
<td>NH₄/Charcoal</td>
<td>1:500</td>
<td>1.0E-04</td>
<td>1.0E-04</td>
<td>0.997</td>
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<td></td>
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<td>3.0E-05</td>
<td>3.0E-04</td>
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<td>-0.6287</td>
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</tr>
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<td>11738</td>
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<tr>
<td>P/Mg-SCBB</td>
<td>1:500</td>
<td>1</td>
<td>1.0E-04</td>
<td>2.0E-05</td>
<td>0.996</td>
<td>NH₄/Charcoal</td>
<td>1:200</td>
<td>4.0E-04</td>
<td>2.0E-04</td>
<td>0.996</td>
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<td></td>
<td></td>
<td>2</td>
<td>1.0E-04</td>
<td>9.0E-05</td>
<td>0.710</td>
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<td></td>
<td>3.0E-05</td>
<td>4.0E-04</td>
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</tr>
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<td></td>
<td></td>
<td>3</td>
<td>-0.8975</td>
<td>10281</td>
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<td>0.834</td>
</tr>
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<td></td>
<td>4</td>
<td>-0.8195</td>
<td>9197.2</td>
<td>0.736</td>
<td></td>
<td></td>
<td>-1.7482</td>
<td>4671.9</td>
<td>0.834</td>
</tr>
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</table>

The total removal percent of ammonium (adsorbed + volatilized) was calculated for the tested sorbents after 12 h and at 1:200 ratios and it was found to follow the order: Zeolite > Mg-SCBB > SCBB > Charcoal (95.89, 89.59, 70.78 and 67.17%, respectively).

Although adsorption of phosphate from aqueous solutions was not observed at all for Zeolite, Charcoal or SCBB, it was only evident for Mg-SCBB. Maximum removal percent of phosphate by Mg-SCBB was reached after 2 h of contact time (Fig. 7). Phosphate removal was higher at 1:200 than at 1:500 ratios (79.88 and 50.01%, respectively) exhibiting the same kinetic trend at the two ratios. Yao et al. (2013) found that Mg-modified biochars were found to have a better ability of phosphate removal from aqueous solutions. They reported that Mg(OH)₂ and MgO nanoparticles were developed on the particles’ surfaces of Mg-modified biochars which served as the sorption sites for aqueous phosphate.

Fitting the sorption data of ammonium and phosphate kinetics models revealed that the pseudo-second-order equation (Eq. 6) was capable of representing the experimental data.
Table 4. Adsorption kinetics parameters calculated from fitting the linearized form of pseudo-second-order equation (Eq. 6-1)

<table>
<thead>
<tr>
<th>Sorbate/sorbent/ratio</th>
<th>Slope</th>
<th>Intercept</th>
<th>R²</th>
<th>qₑ</th>
<th>k</th>
</tr>
</thead>
<tbody>
<tr>
<td>NH₄/SCBB/200</td>
<td>3.0E-04</td>
<td>1.0E-04</td>
<td>0.999</td>
<td>3333.3</td>
<td>9.0E-04</td>
</tr>
<tr>
<td>NH₄/SCBB/500</td>
<td>1.0E-04</td>
<td>1.0E-04</td>
<td>0.997</td>
<td>10000.0</td>
<td>1.0E-04</td>
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<tr>
<td>NH₄/Mg-SCBB/200</td>
<td>3.0E-04</td>
<td>1.0E-04</td>
<td>0.999</td>
<td>3333.3</td>
<td>9.0E-04</td>
</tr>
<tr>
<td>NH₄/Mg-SCBB/500</td>
<td>1.0E-04</td>
<td>4.0E-05</td>
<td>1.000</td>
<td>10000.0</td>
<td>2.5E-04</td>
</tr>
<tr>
<td>NH₄/Zeolite/200</td>
<td>2.0E-04</td>
<td>1.0E-04</td>
<td>0.998</td>
<td>5000.0</td>
<td>4.0E-04</td>
</tr>
<tr>
<td>NH₄/Zeolite/500</td>
<td>1.0E-04</td>
<td>1.0E-04</td>
<td>0.997</td>
<td>10000.0</td>
<td>1.0E-04</td>
</tr>
<tr>
<td>NH₄/Charcoal/200</td>
<td>4.0E-04</td>
<td>2.0E-04</td>
<td>0.996</td>
<td>2500.0</td>
<td>8.0E-04</td>
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<td>NH₄/Charcoal/500</td>
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<td>1.0E-04</td>
<td>0.997</td>
<td>10000.0</td>
<td>1.0E-04</td>
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<tr>
<td>P/Mg-SCBB/200</td>
<td>2.0E-04</td>
<td>2.0E-04</td>
<td>0.991</td>
<td>5000.0</td>
<td>2.0E-04</td>
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<tr>
<td>P/Mg-SCBB/500</td>
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<td>2.0E-05</td>
<td>0.996</td>
<td>10000.0</td>
<td>5.0E-04</td>
</tr>
</tbody>
</table>

Fig. 8. Fitting data of NH₄⁺ and P adsorption by SCBB and Mg-SCBB to the linear form Eq. 6-1 of the pseudo-second-order kinetic equation
Fig. 9. Fitting data of NH$_4^+$ and P adsorption by Zeolite and Charcoal to the linear form Eq. 6-1 of the pseudo-second-order kinetic equation

Fig. 10. Fitting data of NH$_4^+$ adsorption on the tested sorbents to A) Freundlich, and B) Langmuir models
Table 5. Calculated parameters of Freundlich model from the corresponding linear regressions

<table>
<thead>
<tr>
<th>Sorbate/sorbent</th>
<th>slope</th>
<th>intercept</th>
<th>R²</th>
<th>1/n</th>
<th>Kf</th>
</tr>
</thead>
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<tr>
<td>Ammonium</td>
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</tr>
<tr>
<td>NH₄/SCBB</td>
<td>0.895</td>
<td>3.099</td>
<td>0.982</td>
<td>0.894</td>
<td>1255.5</td>
</tr>
<tr>
<td>NH₄/Mg-SCBB</td>
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<td>3.411</td>
<td>0.913</td>
<td>0.756</td>
<td>2573.9</td>
</tr>
<tr>
<td>NH₄/Zeolite</td>
<td>0.636</td>
<td>3.377</td>
<td>0.872</td>
<td>0.636</td>
<td>2379.6</td>
</tr>
<tr>
<td>NH₄/Charcoal</td>
<td>0.510</td>
<td>3.465</td>
<td>0.911</td>
<td>0.510</td>
<td>2920.1</td>
</tr>
<tr>
<td>Phosphate</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>P/Mg-SCBB</td>
<td>0.478</td>
<td>3.602</td>
<td>0.982</td>
<td>0.478</td>
<td>4002.2</td>
</tr>
</tbody>
</table>

Fig. 11. Fitting data of P adsorption on Mg-SCBB to A) Freundlich, and B) Langmuir models

Regression parameters of fitting ammonium and phosphate adsorption data are listed in Table 3. Plotting the values of 1/q vs. t had the highest determination coefficient (R²) of the linear regression plots for all the tested sorbents and ratios (Fig. 8 and 9). Therefore, the linearized form Eq. (6-1) of the pseudo-second-order equation was the best fit. This may suggest the intraparticle diffusion controlled adsorption of ammonium and phosphate on the tested biochars (Ghasemi et al., 2013; Plazinski et al., 2013; Zhang et al., 2015). Parameters of adsorption kinetics (qₑ and k) were then calculated using the obtained slope and intercepts of Eq. (6-1). Generally, the adsorption capacity of ammonium from the aqueous solutions by SCBB and Mg-SCBB were similar and it was higher at 1:500 than at 1:200 (10000 and 3333.3 mg NH₄⁺ kg⁻¹, respectively). The same trend of qₑ was found for phosphate adsorption by Mg-SCBB which recorded 5000 and 10000 mg PO₄³⁻ kg⁻¹ at 1:200 and 1:500, respectively.

Adsorption isotherms:

Data of ammonium adsorption by the tested sorbents at different concentrations showed that adsorption of ammonium increases with increasing its initial concentration in the aqueous solution for all sorbents. Fitting these data to different isotherm models revealed that Freundlich isotherm model was the best fit model (highest R²) to describe the experimental data (Fig. 10) followed by Langmuir model (R² from 0.624 to 871). The parameters of Freundlich model were calculated from the regression equations obtained (Table 5). The calculated parameters may indicate that adsorption capacity (Kₐ) of sorbents for ammonium followed the order: Charcoal> Mg-SCBB> Zeolite> SCBB (Table 5). However, the intensity parameter (1/n) had the range...
from 0.510 to 0.894 and followed the order: SCBB > Mg-SCBB > Zeolite > Charcoal. The calculated n values ranged from 1.96 to 1.12 (between 1 and 10). This may indicate the favorable adsorption of ammonium by the all tested sorbents (Hafshejani et al., 2016).

Isotherms of phosphate adsorption by Mg-SCBB (Fig. 11) confirm that adsorption of phosphate on Mg-SCBB was affected by phosphate concentration. These data were better fitted to the Freundlich model than the Langmuir model (R²: 0.97 and 0.89, respectively). The calculated phosphate adsorption capacity and intensity of this sorbent were 4002.2 and 0.478 mg kg⁻¹, respectively. The calculated value of n (2.09) may indicate the favorable adsorption of phosphate ion on Mg-SCBB. These results are consistent with those obtained by Shwantes et al. (2015). On the other hand, Trazzi et al. (2016) found that Langmuir model gave higher R² than Freundlich model for adsorption of phosphate ions on sugarcane bagasse biochar.

Accordingly, SCBB and Mg-SCBB biochars proved to have the ability to adsorb ammonium from aqueous solutions compared with the well-known sorbents Zeolite and Charcoal. In addition, Mg-SCBB was the only sorbent capable of removing both ammonium and phosphate from these solutions. Therefore, Mg-SCBB can be used for an efficient dual removal of these two ions from aqueous solutions.

CONCLUSIONS

Pyrolysis of sugarcane bagasse at 500 °C under oxygen-limited condition produced a biochar which is capable of efficiently removing ammonium ions from aqueous solutions and it was comparable to commercial Charcoal and Zeolite sorbents. Pre-pyrolysis MgSO₄ impregnation of bagasse feedstock produced modified biochar with a dual capability of removing ammonium and phosphate ions from aqueous solutions. Changes in the physical and chemical properties occurred in the generated biochars as compared to the feedstock. Analysis of FTIR bands and SEM investigations revealed that surface chemical functional groups and meso- and micropores were developed due to pyrolysis. Adsorption kinetics data of both ammonium and phosphate ions on the tested sorbents were best described by the linearized form of the second-pseudo-order equation indicating the intraparticle diffusion controlled adsorption of ammonium and phosphate. Ammonium and phosphate adsorption isotherms were best fitted to the Freundlich model and revealed that the adsorption of these ions was favorable by the generated biochars. The Mg-modified sugarcane bagasse biochar can be considered as a promising sorbent for dual removal of ammonium and phosphate ions from aqueous solutions. Further research is needed to investigate the influences of various physical and chemical factors on the efficacy of this biochar in remediating contaminated wastewater.

REFERENCES


المملوک العربي

استخدام مخلفات عصر قصب السكر المعدلة بالمغسوب في الإزالة المزدوجة لأيونات الأمونيوم والفسفات من المحلول المائية

مهير السيد صالح، رامي مرسى رقية

على إزالة أيونات الفوسفات من المحلول المائية. وكان
مقدار إزالة أيونات الأمونيوم والفوسفات عند نسبة مادة
الحوز إلى الحلول 20000، بشكل عام أعلى من
النسبة 5000:1 عند نفس التركيزات. وقد ثبت حدوث تغليف
لفحام الأمونيوم من المحلول أثناء عملية الإحراق
نتيجة ارتفاع رقم pH لمحلول مواد الإحراق
المختبر مع المحلول.

Adsorption Kinetics
وجد أن نتائج حركة إحراق
أيونات الأمونيوم والفوسفات تنطبق مع معادلة شبيهة
الدرجة الثانية، Pseudo-Second-Order، بما يدل على أن
ميكانيكا الإحراق في حلبات مادة الإحراق هي
الخطوة الحاسمة في عملية الإحراق، كما ثبت أن
منحنى الإحراق عند درجات حرارة متساوية
يظهر ب nhiễعًا أفضل مع نموذج
Adsorption Isotherms، وتأتي في
مقياس مختلفة (SCBB، Mg-SCBB).
FMCD، ونماذج أخرى من التحليل
الروسيكبي الرياسي، ونماذج SEM، وكذلك بعض الخواص
الفيزيائية والأيضية للفحم الناتج على ظهور العديد
من المفاعلات الكيميائية الطبيعية، وتكوين المسام المتوزعة
والدقيقة، وزراعة ساحة الأسفلت، وكذلك ارتفاع السعة
التلبية الكاتيونية بالمقارنة بالمادة الأولية (SCBB).

الذان تم
مقدار Mg-SCBB ونماذج
مقدار إنتاج وقود وقود على إحراق أيونات الأمونيوم
Charcoal والزيلوليت Zeolite. كما وجد أن الفحم
Mg-SCBB من تخسيب مصاصة القصب بالمغسوب) هو الوحيد الذي

扸

SCBB مصاصة القصب الخام (Mg-SCBB) بطريقة
التمثيل الحراري عند درجة حرارة 600 °C تحت ظروف
محددة من الأمورين لمصاصة القصب الخام والمحمية
بكراتي المغسوب، على التوالي. وقد دلت تحايل
منحنين، وفضلات RSIR، وفوتو-FTIR
بالميركوبك الرياسي، وذلك ببعض الخواص
الفيزيائية والكيميائية. وللقصة المحايدة على ظهور العديد
من المفاعلات الكيميائية الطبيعية، وتكوين المسام المتوزعة
والدقيقة، وزراعة ساحة الأسفلت، وكذلك ارتفاع السعة
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الذان تم
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扸