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Removal of phosphate ion from water using chemically modified biomass of sugarcane bagasse

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----------ABSTRACT----------
The sugarcane bagasse was chemically modified method, with epichlorohydrin and triethylamine in the presence of ethylenediamine and N,N-dimethylformamide. The characterization of chemically modified sugar cane bagasse was measured by using SEM and FTIR analysis. Batch experiments were conducted to study the adsorption property of phosphate from aqueous solutions. The results of characterization of chemically modified sugar cane validated the increased amine groups in MSB and its maximum sorption capacity Q_{max} of phosphate was found 1.05 mmol g^{-1}. Based on this study, the thermodynamic parameters like standard Gibb's free energy (ΔG°), standard enthalpy (ΔH°) and standard entropy (ΔS°) were evaluated. Langmuir isotherm was well fitted isotherm in the temperature range studied. The results show that the chemically modified sugar cane bagasse can be efficiently used for the removal of phosphate ion from water as a low cost alternative compared to commercial activated carbon and other adsorbents reported to check the eutrophication.

KEYWORDS: *Phosphate ion; Adsorption; Activated sugar cane bagasse; Kinetics; Thermodynamics; Wastewater treatment*

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I. INTRODUCTION

The production of large-scale wastewater is unavoidable consequence of modern-day societies. The wastewater generated is hazardous to human health and the environment. Domestic and agro-industrial wastewater release large amount of phosphorus into wastewater and these nutrients are directly responsible for eutrophication. Thus, proper wastewater treatment and disposal should be practiced to reduce the possibility of water pollution.Removal of phosphate from wastewater prior releasing to the environment had done by many researcher and they used several methods including chemical and biological methods such as reverse osmosis, (Jeppesen, Shu et al. 2009) and (Greenlee, Lawler et al. 2009), electro-dialysis (Mohamed 2002), biological de-nitrification (Uygur and Kargı 2004), and adsorption (Fernández-Olmo, Fernández et al. 2007). Further investigation has been done on adsorption, which focused on the preparation of adsorbent by using agricultural waste (Orlando, Baes et al. 2002), (Wang, Gao et al. 2007) and (Xu, Gao et al. 2010).The use of lignocellulosic agricultural waste (AW) in the preparation process seems as a potential way to obtain novel and low cost anion adsorbent. Other than that, the proper use of agriculture-based adsorbent will prevent the use of petroleum derivatives as raw material and avoid water pollution by hazardous substances (Orlando, Okuda et al. 2003). This is due to the large amount of anion exchange based adsorbent could be generated from high volume, low-cost agricultural by-product as starting materials that contain various amount of cellulose and lignin (Wartelle and Marshall 2006).This project illustrates the preparation of an adsorbent from sugarcane bagasse (SB) by using ETM (epichlorohydrin-triethylamine method). The main objective of this paper is to study the phosphate sorption capacities of the modified sugarcane bagasse (MSB). In this project, batch sorption experiments were performed to evaluate the effect of MSB dosage and temperature on the phosphate removal. The characteristics of MSB and its property for phosphate removal were studied using various characteristic measurements.

II. MATERIALS AND METHODS

Chemicals and reagents : Triethylamine and epichlorohydrin from Sigma-Aldrich, ethylenediamine from QRëC, and N,N-dimethylformamide from Fluka were used in the modification process in this experiment. In this study PhosVer 3 from HACH was used to develop the color with phosphate ions.

Sugarcane bagasse preparation : Sugarcane bagasse (SB) was used as the starting material in this experiment. The raw SB was obtained from street vendor at Sungai Dua Night Market, Penang. The SB was soaked and washed with distilled water. It was subsequently washed with water until odorless. The washed SB was dried in

the oven at 60 °C for 24 h to achieve its constant weight. Then, it was grind into powder form and the grinded SB was sieved obtained resulting material with diameters from 100 to 250 µm.

Preparation of modified sugarcane bagasse : Ten grams of powdered SB was weight crucially and it is used to react with 20 ml of epichlorohydrin and 25 ml of N,N-dimethylformamide. The mixture was stirred for 60 min and the temperature was maintained at 80 ºC (Xu, Gao et al. 2010). The process was followed by adding 4 ml of ethylenediamine and the mixture was stirred for 45 min at 85 ºC. An aliquot of 20 ml of triethylamine was added and the mixture was stirred for 120 min at 85 °C. The resulting product was washed with 500 ml of distilled water to remove the residual chemicals (Xu, Gao et al. 2009). The washed MSB was dried in the oven at 60 °C for 12 h until the constant weight achieved. The dried MSB was sieved to obtained particles smaller than 250 µm in diameter and then used in all the adsorption experiments.The synthetic reactions of MSB using SB as a starting material are shown in Fig. 9 where cellulose is considered as targeting molecule to start the reaction. The reaction between epichlorohydrin and cellulose was induced after the hydroxyl groups in the cellulose molecule activated, producing hydroxyl cellulose ether. The hydroxyl cellulose ether was then cyclized by the catalyst existing in the alkaline condition to produce the epoxy cellulose ether that was used as the intermediate in the reaction. The MSB was obtained after the graft reaction between epoxy cellulose ether and triethylamine (Xu, Gao et al. 2009).

Characteristic of MSB

Scanning Electron Micrograph : The morphology and structural properties of the MSB and SB were analyzed using a field-emission scanning electron microscope (FE-SEM) (LEO SUPRA 55V, Carl Zeiss, Oberkochen, Baden-Württemberg, Germany) with an operating voltage of 5 kV. The SEM process was conducted at School of Biology, Universiti Sains Malaysia (USM). The sample were prepared by mounted it on specimen stub and coated it with an ultrathin coating of gold, deposited on the sample by low-vacuum sputter coating. The SEM measurements were done to investigate the structures of MSB and SB. The micrographs of the sample were taken with magnification of 500 X, 1.00 K X, 3.00 K X, and 5.00 K X for both MSB and SB.

FT-IR analysis : IR spectra were recorded on a Thermo / Nicolet Avatar 360 spectrometer in the 4000-400 cm^{-1} region. The FT-IR spectrometer was linked to a computer loaded with the IRDM (IR Data Manager) program to process the recorded spectra. The samples (MSB or SB) were grind with potassium bromide finely, to remove scattering effects from large crystals. This powder mixture is then pressed in a mechanical press to form a translucent disc through which the beam of the spectrometer can pass. The IR spectrums of the samples were recorded. FT-IR analysis was done in order to investigate the functional groups those were present in MSB and SB.

Phosphate removal experiment : In the following experiments, MSB dosage and temperature were selected as experimental parameters to study their impact on the MSB and SB adsorption ability. To study the impact of MSB dosage on the phosphate removal in aqueous solution, different quantities (0.025, 0.05, 0.075, and 1.00 g) of MSB were placed in 100 ml Erlenmeyer flask with 50 ml of 50 mg/L KH_2PO_4 solution at 20 ± 2 ºC and then shaken at 120 rpm for 80 min using incubator shaker. The phosphate concentration was determined spectrophotometrically by Ammonium-molybdate colometric method (PhosVer 3 Phosphate Reagent) and the absorbance was determined using a UV-VIS spectrophotometer at an absorbance wavelength of 700 nm.

To determine the effect of temperature on the sorption process, the experiments were conducted at three different temperatures (20, 40, 60 ºC). An aliquot of 0.1 g of MSB was immersed in 100 ml Erlenmeyer flasks with 50 ml of KH_2PO_4 solutions in different concentrations (25-500 mg (P) L^{-1}) and shaken at 120 rpm for 80 min using incubator shaker at 20 ± 2 ºC. The phosphate concentration was determined spectrophotometrically by Ammonium-molybdate colometric method (PhosVer 3 Phosphate Reagent) and the absorbance was determined using a UV-VIS spectrophotometer at an absorbance wavelength of 700 nm. The procedure was repeated using different temperature (40 and 60 ºC). The calibration curve was constructed to determine the phosphate concentration based on the absorbance reading. From the graph, $y = 0.338X + 0.208$ with $R^2 = 0.9973$ was obtained.

III. RESULT AND DISCUSSION

Characteristic of MSB

SEM analysis: The results of SEM measurements in the structures of MSB and SB are shown in Fig. 1- 10. The surface of SB became smooth and the impurities on the surface decreased after pretreatment as clearly shown in Figure 5-8. The resulting result is due to the removal of impurities on the surface of SB during the pretreatment process (Ma, Li et al. 2011). During that process, the SB was washed with water for several times to remove

impurities such as, debris, dust, and small granite. However the SB does not further treated and was used as the control in this study. Thus, tighter surface and less porous structure can be observed at SB compared to MSB structure. Irregular surface and porosity is important in facilitating adsorption process. This feature favors the adsorption of phosphate from aqueous media. The surface of MSB became rough as shown clearly in Figure (1-4). The resulting morphology of the MSB was due to the graft copolymerization reaction with organic monomer. It indicated that after the reaction, the organic monomers were grafted in the cellulose skeleton, which resulted in a broad network and increased porous structure on the surface of the product. The increase of micro porous gaps on the MSB surface will lead to a substantial increase of adsorption capacity. This is because, aqueous medium can easily diffused into the MSB through the micro porous gaps (Liu, Miao et al. 2009; Ma, Li et al. 2011). This modified feature favors the adsorption of phosphate from water.

Based on the SEM analysis from both MSB and SB, it clearly shown a significant differences between modified and unmodified SB. Thus, this is proven that the chemical reactions successfully modified the SB and subsequently the morphology of the adsorbent, which is important in facilitating the adsorption process (Alomá, Martín-Lara et al. 2012).

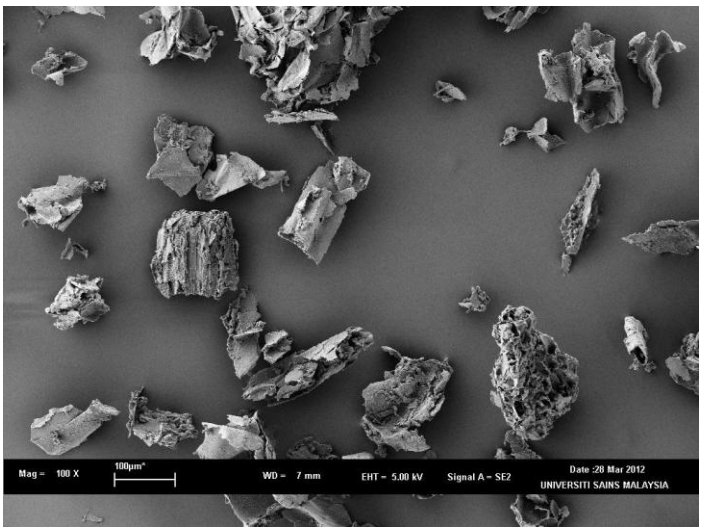

Figure 1: SEM of MSB for magnification 100 X

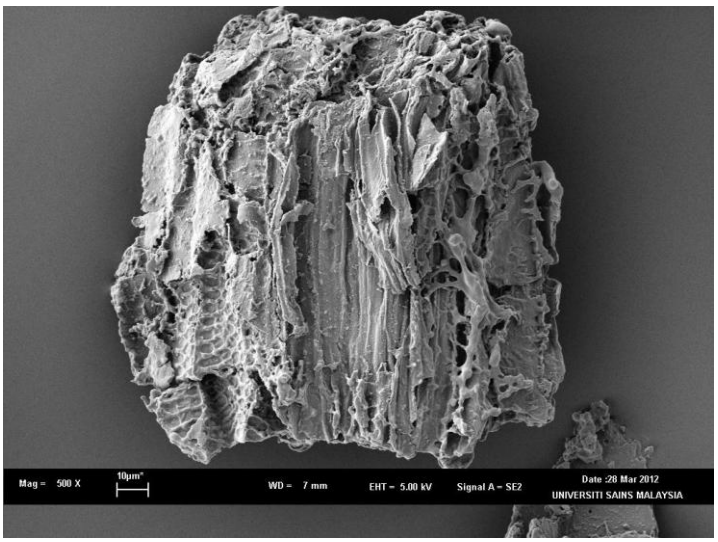

Figure 2: SEM of MSB for magnification 500 X

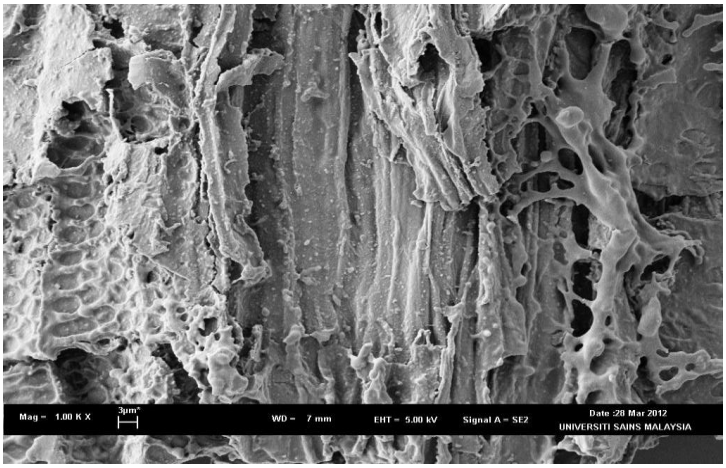

Figure 3: SEM of MSB for 1.00 K X

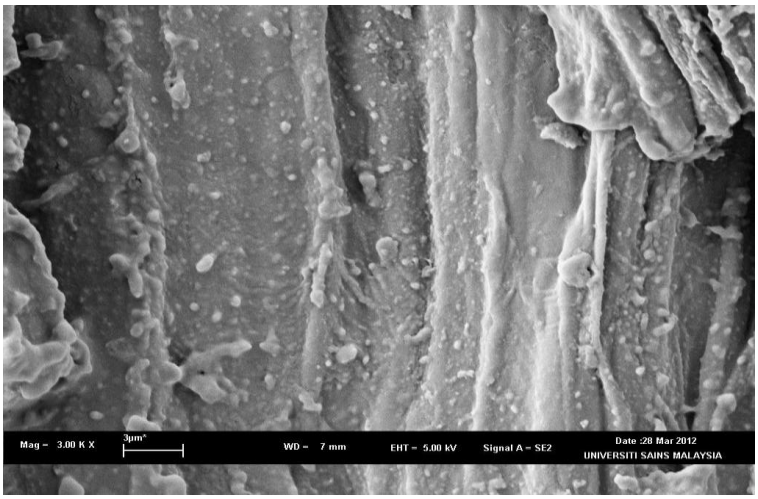

Figure 4: SEM of MSB for magnification 3.00 K X

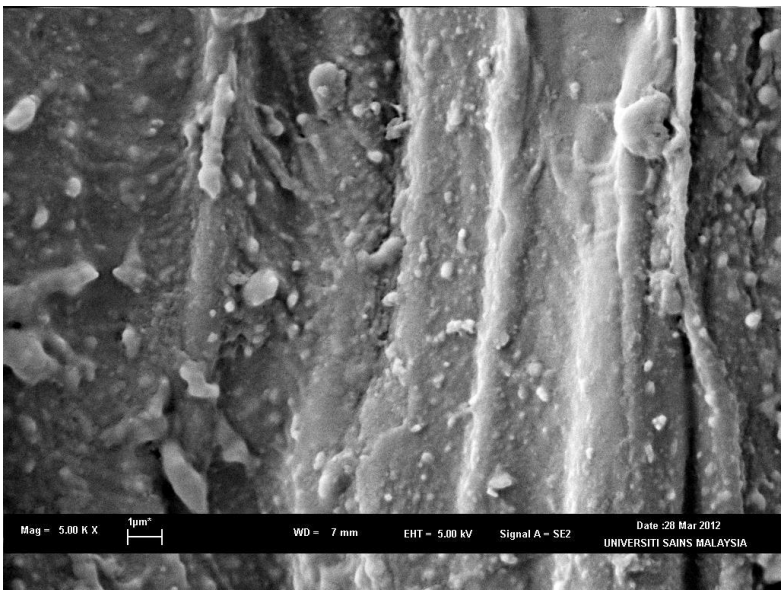

Figure 5: SEM of MSB for magnification 5.00 K X

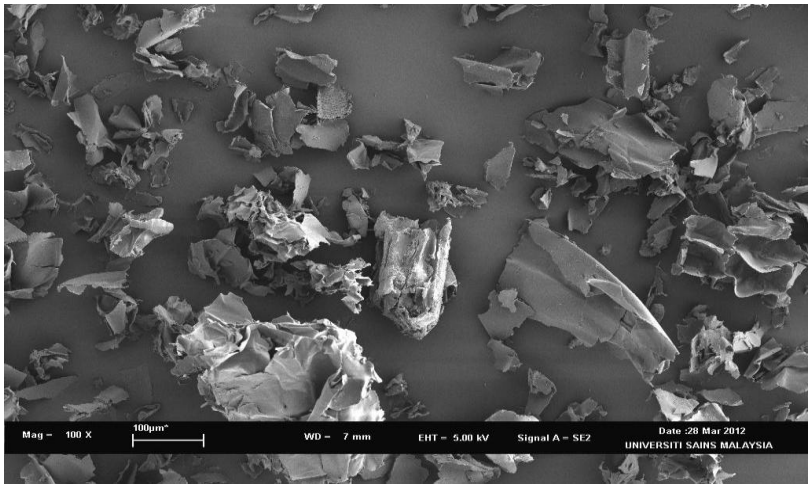

Figure 6: SEM of SB for magnification 100 X

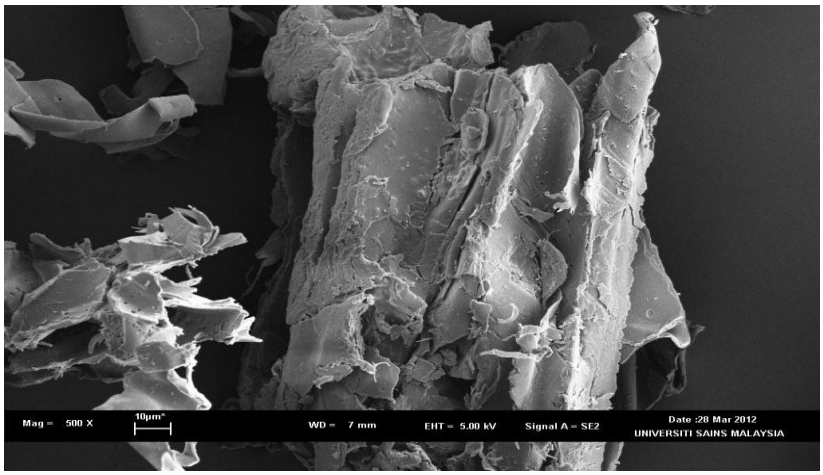

Figure 7: SEM of SB for magnification 500 X

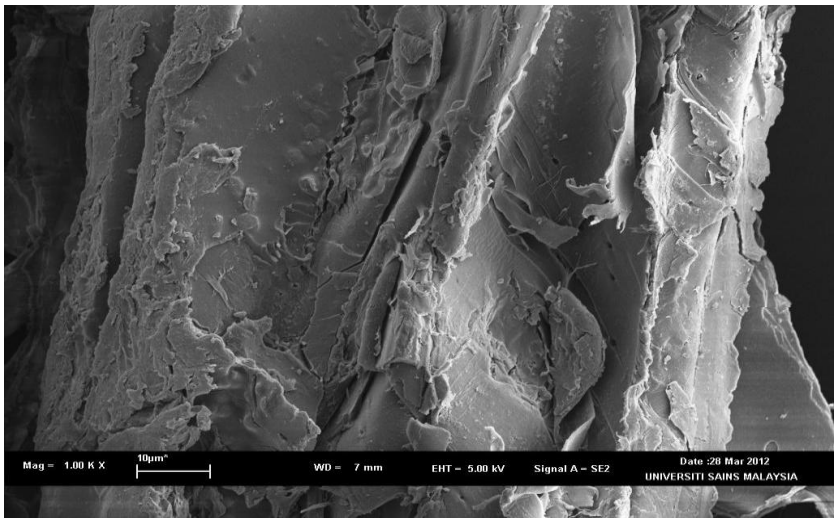

Figure 8: SEM of SB for magnification 1.00 K X

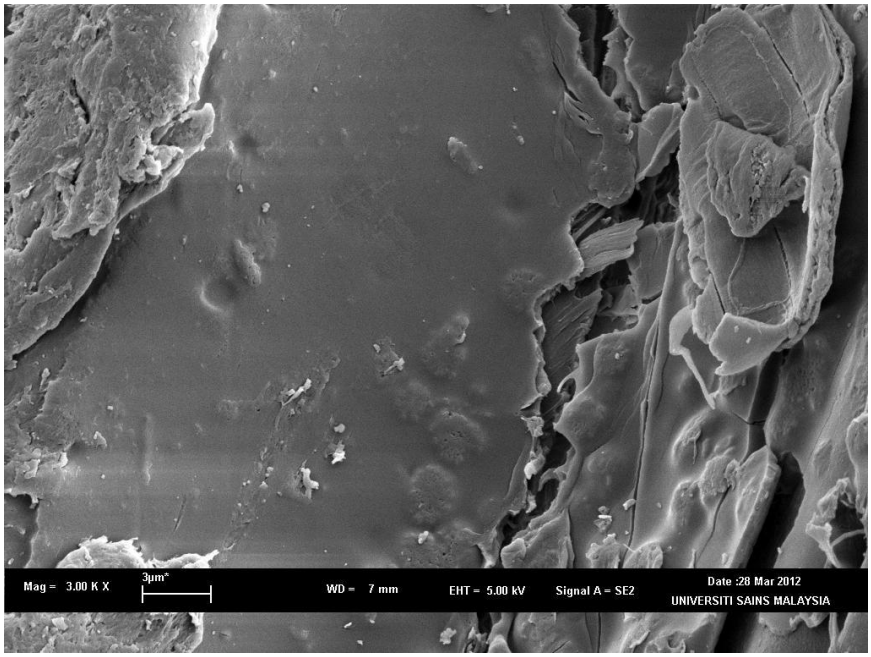

Figure 9: SEM of SB for magnification 3.00 K X

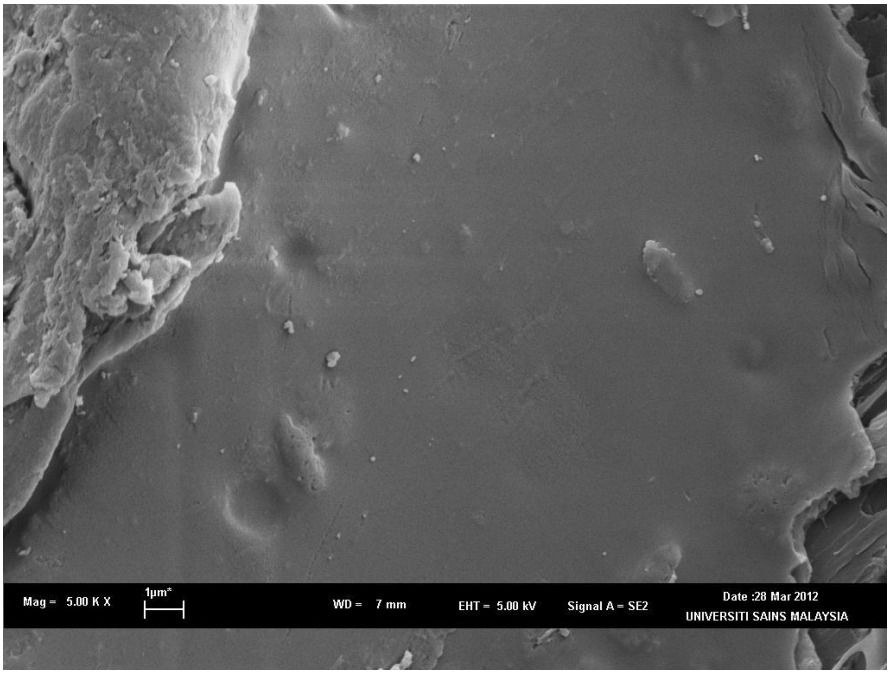

Figure 10: SEM of SB for magnification 5.00 K X

FT-IR analysis of MSB : The FT-IR study showed some important changes in some bands of SB and MSB. For the SB, a large vibration of the band at 3411.20 cm^{-1} proves the existence of hydroxyl groups in the SB (Xu, Gao et al. 2009; Xu, Gao et al. 2010). The peak at 2919.24 cm^{-1} is the associated with the special vibration of C-H aliphatic in SB (Xu, Gao et al. 2010). For the SB without modification, which possesses cellulose with a significant amount of lignin, these bands are clearly evident in a spectrum of higher resolution. Other important but less intense bands at about 1511 cm^{-1}, 1457 cm^{-1}, and 1427 cm^{-1} attributed to aromatic ring vibrations with some methoxy group substituent (C-O stretching) in syringyl (S) and guaiacyl (G) units present in the lignin. The bands at 1375 cm^{-1} refer to stretching of C-H of acetyl groups from hemi-cellulose esters. The C-O vibrational stretching of the syringyl (S) in lignin units occurs at 1327 cm^{-1}. At 1250 cm^{-1} stretching of the C-O from acetyl groups present on the bagasse fibres occurs (Carvalho, Martins et al. 2011).

The carbonyl stretching from ester groups can be seen at 1163 cm^{-1} and is characteristic of p-hydroxyphenyl groups in grassy structures, which are present in significant quantities in bagasse fibers. The graft copolymerization of organic monomer on surface of sugarcane bagasse led to important modifications in the FT-IR spectrum. In contrast to the FT-IR analysis of SB, the increase of various groups was observed in the FT-IR analysis of MSB. The spectra of MSB (Figure 12) showed new strong bands at 1657 cm^{-1} corresponding to the presence of amide and amine functions, and one band at 1507 cm^{-1} corresponding to the angular deformation of the N-H bond of the amide function. The band at 1163 cm^{-1} corresponds to the asymmetric stretch of C-N-C bond (Karnitz Jr, Gurgel et al. 2007).Modification also occurred in the band around 1048 cm^{-1}, attributed to the C-O vibrational stretching of the first order aliphatic C-OH and the ether C-O-C linkages, present in the cellulose and lignin structures. The modifications were principally related to the shape and intensity of the bands, but no significant change in their position was observed. This may be a consequence of the longer distance between the adsorption sites for both lignin and cellulose, with respect to the ether C-O-C bonds (Carvalho, Martins et al. 2011). As a result of the FTIR analysis, the adsorption of phosphate onto MSB can be ascribed to the significant increase of amino groups which are used as adsorption sites for phosphate removal (Gao, Xu et al. 2009; Xu, Gao et al. 2009).

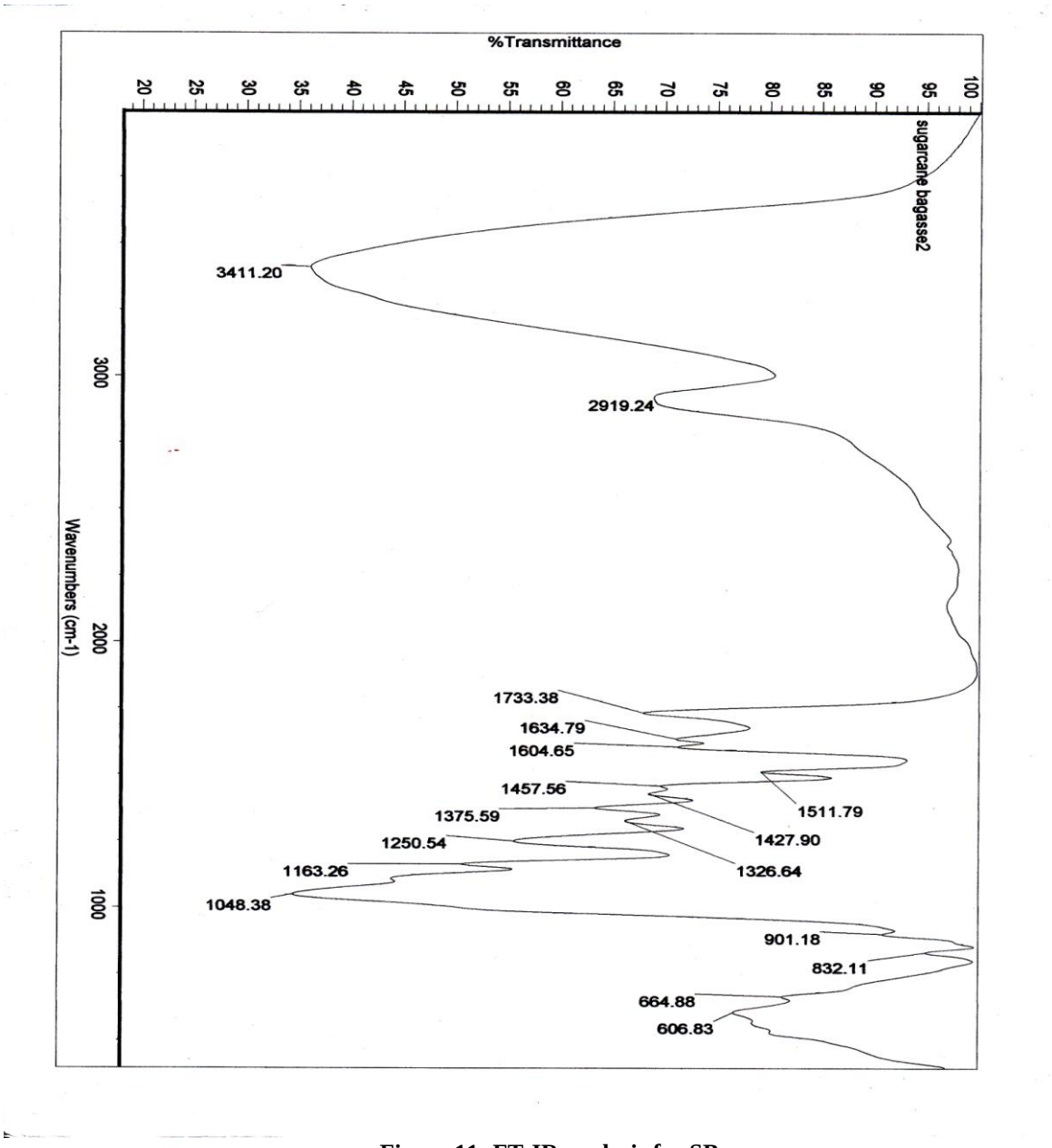

Figure 11: FT-IR analysis for SB

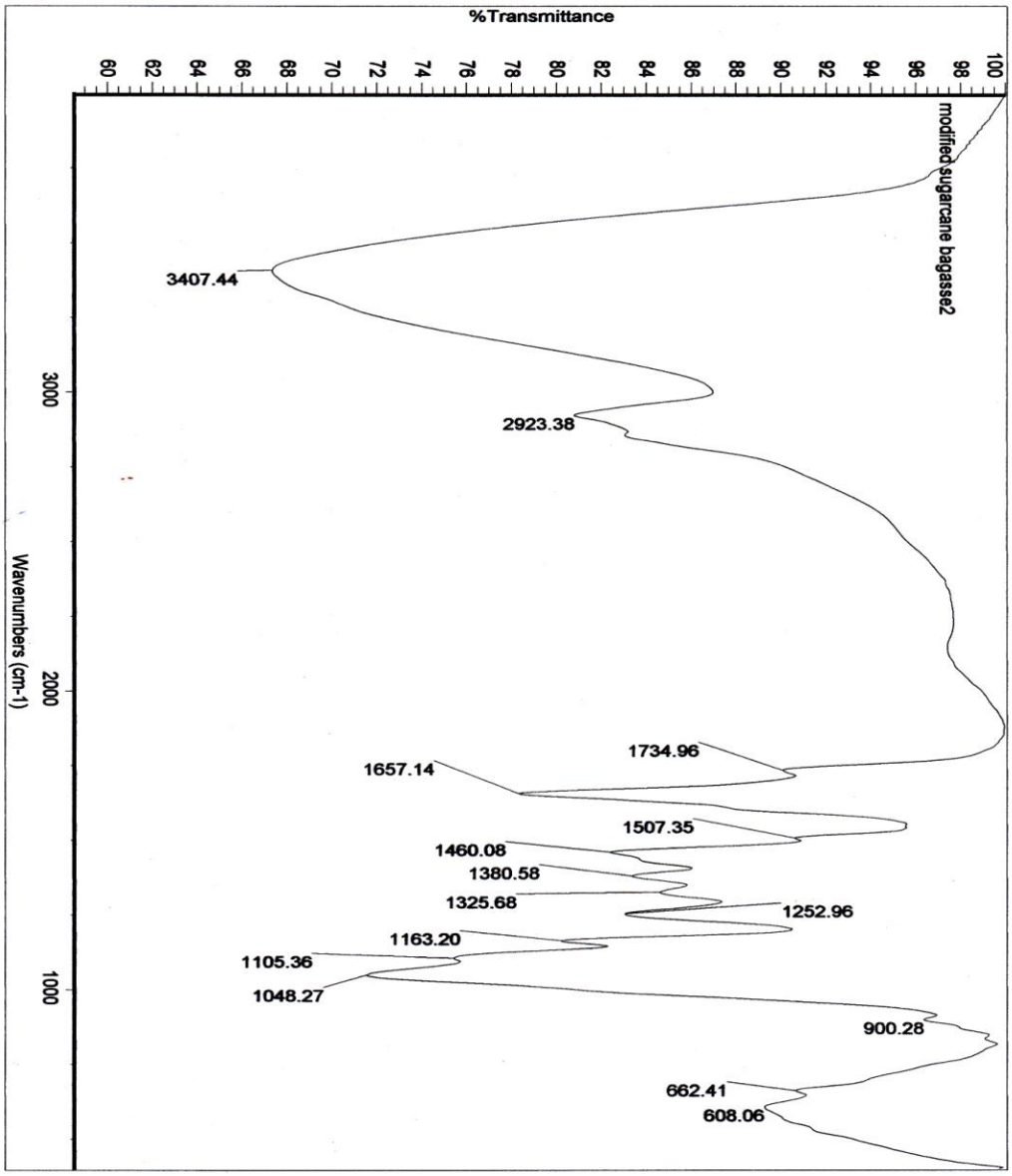

Figure 12: FT-IR analysis for MSB

Effect of adsorbent dosage : MSB dosage is an important parameter because it determines the capacity of an anion exchanger for a given initial concentration of the phosphate under operating conditions. The effect of MSB dosage on the sorption of phosphate is shown in Figure 13. The removal efficiency of phosphate increase significantly from 54.1% to 95.2% with increasing MSB dosage from 0.5 to 2 g/L, and the adsorption is nearly constant when dosage exceeds 2 g/L. With increasing adsorbent dosage, more adsorption sites are available for sorption thus more phosphate will be remove (Xu, Gao et al. 2009). Nearly all the phosphate is adsorbed when the adsorbent is increased to 2 g/L consequently; no increasing in the removal efficiency was observed. When the phosphate removal reaches near 100%, addition MSB added would not be used (Xu, Gao et al. 2010). Thus, it is reasonable that the phosphate removal should not increase much when the dosage of MSB is higher than 2 g/L.

Figure 13 also shows the phosphate removal capacity of untreated SB. The low phosphate removal (7.8%) of unmodified SB indicates that the unmodified SB has little effect on sorption of phosphate. The low sorption of phosphate due to adsorption of unmodified SB is only based on surface adsorption (Xu, Gao et al. 2009). Based on the morphology of unmodified SB (Fig. 13), it also proven that a chemical modification is

required to introduce some functional groups into the structure of SB and to increase the porosity of SB structure in order to facilitate the phosphate removal process (Ma, Li et al. 2011).

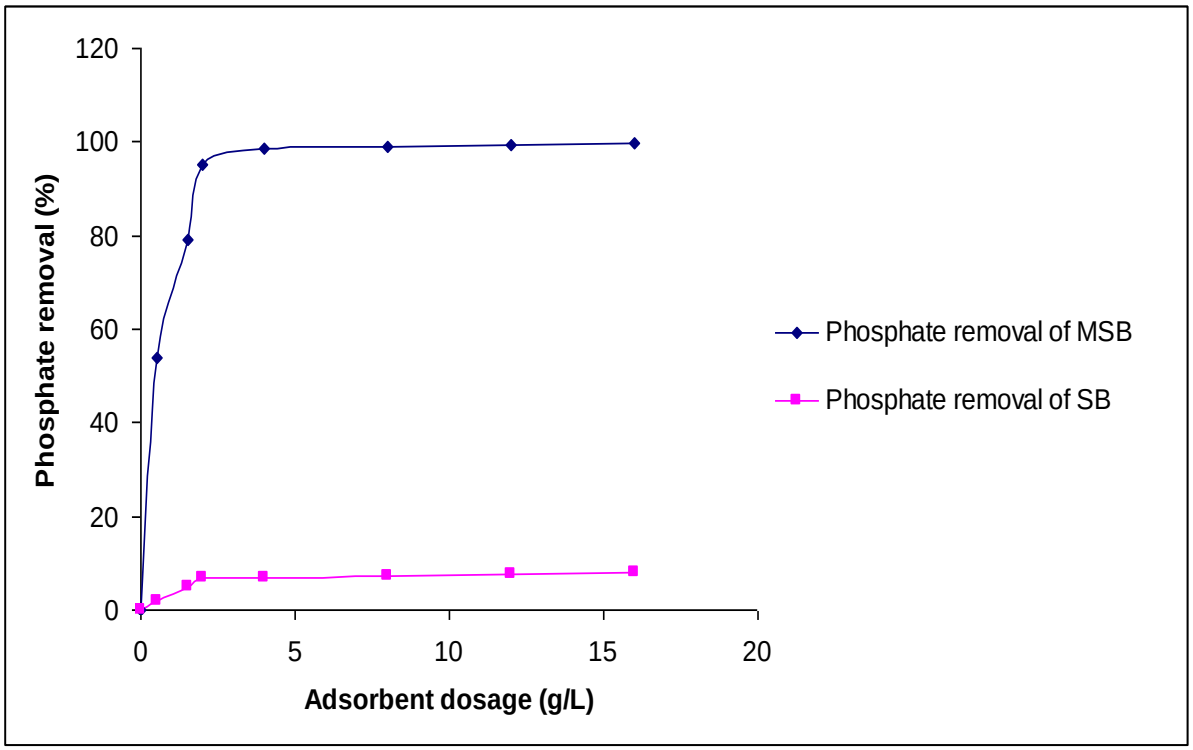

Figure 13: Effect of adsorbent dosage on adsorption of phosphate by MSB and SB (initial $H_2PO_4^-$: 50 mg/L; temperature: 20ºC; shaker speed 120 rpm).

Effect of temperature on adsorption : The effect of temperature on the phosphate adsorption is also shown in Figure 14. The adsorption of the phosphate onto MSB followed at three temperatures, 20, 40 and 60 ºC shows a fast phosphate uptake and a decrease in amount of adsorption when increasing the temperature. The observed decrease in the adsorption capacity with an increase of the temperature from 20 to 60 ºC indicates that lower temperatures are favorable to phosphate removal by adsorption onto MSB. This may be due to a tendency for the phosphate ions to escape from the solid phase to the bulk phase with an increase in the temperature of the solution (Xu, Gao et al. 2009). The experiment data are analyzed by the Langmuir models and thermodynamic models and the results shown is Table 2 and 3.

Langmuir model : Adsorption equilibria can be correlated by means of numerical quantities as the usual Langmuir relationship (Riegel, Tokmachev et al. 2008). Langmuir model is an indication of surface homogeneity of the adsorbent and adsorption phenomena (Wang, Gao et al. 2007). The maximum sorption capacity (Q_{max}) is evaluated by Langmuir isotherm equation in the linear from:

$$\frac{C_e}{q_e} = \frac{1}{K_L q_m} + \frac{1}{q_m} C_e \qquad (1)$$

The sorption isotherms of phosphate by MSB at 20, 40 and 60 ºC are illustrated in Figure 14. Based on the observation in Figure 15 and Table 2, a slight decrease of the maximum sorption capacity with increasing temperature is observed. The high regression correlation coefficient (>0.988) observed in Langmuir isotherm for all temperature indicate that Langmuir isotherm is applicable for describing sorption equilibrium of phosphate (Xu, Gao et al. 2010). The maximum sorption capacity (Q_{max}) for MSB is obtained at 20 ºC, and the value of Q_{max} is 1.05 mmol g^{-1}. Although Q_{max} of MSB is not as high as other modified agriculture wastes (Table 1) such as pine bark (1.06 mmol g^{-1}), rice husk (1.20 mmol g^{-1}) and wheat straw (1.80 mmol g^{-1}) (Xu, Gao et al. 2010), but it is still higher than coconut husk (0.89 mmol g^{-1}), lauan sawdust (0.77 mmol g^{-1}) (Orlando, Baes et al. 2002). The Q_{max} of MSB is also showed much higher than that commercial activated carbon (0.19 mmol g^{-1}) and some anion exchanger resins (0.45-1.36 mmol g^{-1}).

This finding proven that, the ion exchange based adsorbent prepared in this work can be considered as a more efficient and less costly material for phosphate removal in aqueous solution (Orlando, Okuda et al. 2003) (Orlando, Baes et al. 2002). The differences values of Q_{max} between these modified AW are depend on the amount of α-cellulose and lignin present in the AW. This is because; α-cellulose and lignin are important sources of relatively easily accessible hydroxyl unit that can use for the attachment of several functional groups (Orlando, Okuda et al. 2003). Thus, the increase in the lignin content of the AW will resulted in the increased Q_{max}.

Table 1: Q_{max} for modified agriculture based adsorbent and commercial adsorbent (Orlando, Baes et al. 2002; Orlando, Okuda et al. 2003; Xu, Gao et al. 2010)

Adsorbent	Q_{max} (mmol/g)
Wheat straw	1.80
Pine bark	1.06
Rice husk	1.20
Coconut husk	0.89
Lauan sawdust	0.77
Activated carbon	0.19
Anion exchange resin	0.45-1.36

Table 2: Isotherm parameter for phosphate adsorption onto MSB

T (K)	Langmuir			
	Equation	Q_{max} (mmol g^{-1})	K_L (L/g)	R^2
20	$C_e/q_e = 0.0304C_e + 0.5417$	1.05	1.8	0.988
40	$C_e/q_e = 0.0363C_e + 0.6229$	0.88	1.62	0.989
60	$C_e/q_e = 0.0417C_e + 0.5748$	0.76	1.45	0.998

Thermodynamic parameters : The thermodynamic parameters such as change in standard free energy (ΔGº), enthalpy (Δ*H*º) and entropy (Δ*S*º) were calculated to clarify the process of sorption. The thermodynamic parameters were calculated via equation (2) and (3) by applying the Langmuir isotherm:

$$\Delta G^0 = -RT\ln(K_L) \quad (2)$$

$$\ln(K_L) = \frac{\Delta S^0}{R} - \frac{\Delta H^0}{RT} \quad (3)$$

The enthalpy change was determined by plotting ln K_L versus 1/T (Pan, Pan et al. 2008).

The negative value of Δ*H*º and Δ*G*º in Table 3 confirms that the process of phosphate adsorption onto MSB is essentially an exothermic and spontaneous one (Li, Yue et al. 2008). The decrease in the value of - Δ*G*º with increasing temperature from 20ºC to 60ºC shows that the sorption process is less favorable at higher temperature (Xu, Gao et al. 2010). The negative value of Δ*S*º shows the stability process with no structural change at solid liquid interface (Akhtar, Bhanger et al. 2006).

Table 3: Thermodynamic parameter for phosphate adsorption onto MSB

T (K)	Δ*G*º (KJ/mol)	Δ*H*º (KJ/mol)	Δ*S*º ($Jmol^{-1}$ K)
293	-10.87	-17.63	-15.82
313	-10.33		
333	-10.21		

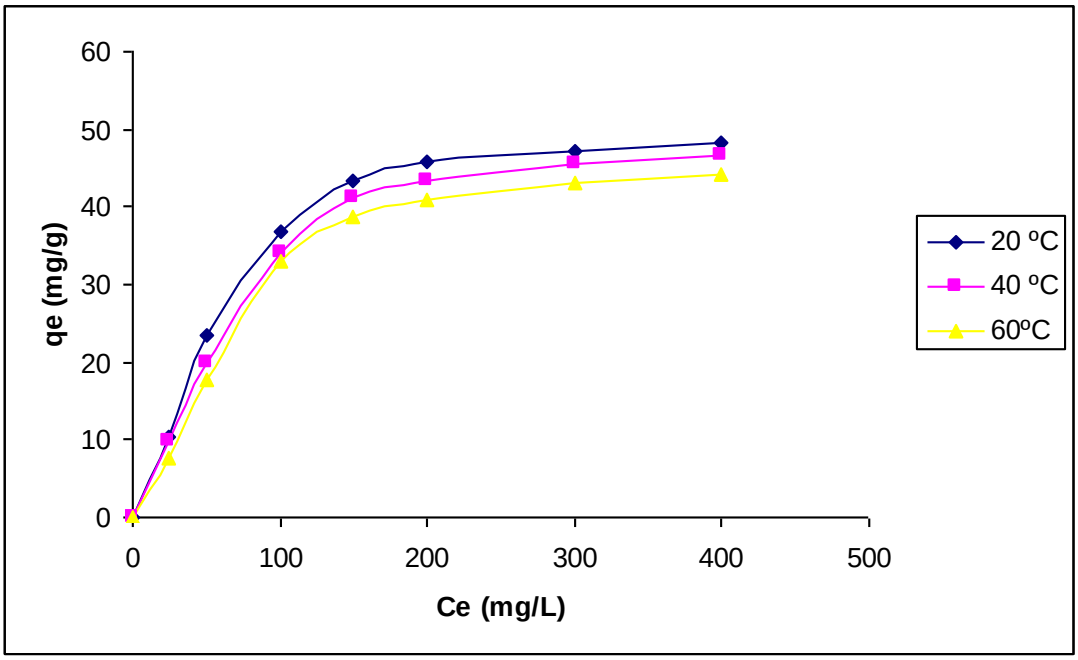

Figure 14: Effect of temperature on sorption isotherm of phosphate by MSB (MSB dosage: 2 g/L; shaker speed = 120 rpm)

IV. CONCLUSION

For this study, it has been proven that MSB can be prepared from raw SB by treating it with epichlohydrin and triethylamine in the presence of ethylenediamine and N,N-dimethylformamide in fixed condition. The characteristic of the MSB can be determine by chemical analysis by using FT-IR to indicated that amine groups were introduced into the framework of MSB. The physical characteristic of the MSB was determined by SEM analysis. From the result of SEM, it was proven that the surface and morphology of the MSB have been modified. Results of batch sorption tests showed that MSB was efficient and an alternative used in an anion exchanger for phosphate removal in aqueous solutions. The optimal phosphate removal was obtained at 2 g L^{-1} of MSB dosage and at temperature of 20 ºC. Evaluation of equilibria was made by Langmuir approach. The Q_{max} estimated by Langmuir isotherm was 1.05 mmol g^{-1}. It can be also concluded that the MSB is an effective and alternative adsorbent for the removal of phosphate ions from wastewaters in terms of efficiency, simple preparation, abundant availability, and low cost.

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