Dissolved solids adsorption of freshwater using synthesized bio-foam composite

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Abstract: In this study, samples of freshwater from the river Nile in Egypt were collected and treated using foamed polyurethane (PU)-ground natural fiber bio-composites. This work aims to measure the effectiveness of total dissolved solids and organic contaminates removal using foamed polyurethane /ground coconut fibers, palm fibers, rice and wheat straw bio-composites. Adsorption process in addition to simulate the batch adsorption data were carried out. Effect of bioadsobent dose at contestant pH was investigated. The results of contaminates removals using bio-composites were compared with the commercial powdered activated carbon adsorbent. It is found that low removal efficiency of TDS using PU-ground rice straw bio-composite. Slight removal of total dissolved solids has been recorded using PU-ground coconut fibers and wheat straw bio-composites. PU/ground palm fibers bio-composites adsorbent shows a significant improvement in TDS removal. It is able to remove up to 37% of total dissolved solids contents. Langmuir and Freundlich models were applied using adsorption test data and evaluated. The thermal adsorption of total dissolved solids using ground palm fibers was verified using Langmuir model.
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1. Introduction

In light of the rapid development of chemical, petrochemical, heavy and light industries in all over the world and the associated population rate increase as a result of that, and because potable/industrial water resources are limited, the need for water recycling to produce fresh water to meet that demand becomes vital (Ben Hamouda et al., 2011)

Freshwater could be used as a cooling medium at different stages in industries; however water contents such as heavy metals and micro-organic materials often cause corrosion of pipes and low cooling efficiency, respectively (K.Z. Mohammed et al., 2012) . With the increase and diversities of the industries which have high influence on the surface water pollution, there is a need to develop the adsorbents for different impurities removal from water than the commercial adsorbents (G. Vijaya Lakshmi et al., 2008). Total dissolved solids are considered as one of indicators to measure the water quality specifically the fresh water streams: rivers and lakes. It is a general indicator of the presence of diverse chemical contaminants. The sources of total dissolved solids in fresh water are mainly agricultural and domestic runoff, leaching of soil contamination and industrial discharges. The most common chemicals are calcium, nitrates and phosphates. The chemicals may be few or thousands of soluble cations, anions, molecules or agglomerations (Bojic et al., 2009). Pesticides are one of the most toxic and harmful elements of TDS. Rocks and soils are natural resources of the TDS.

The most common industrial adsorbents are activated carbon and alumina, because they have high surface areas per unit weight (Issabayeva et al., 2007). Activated carbon is produced by pyrolysis of organic matters such as coconut shell, wood, and bone (Chen et al., 1996). Carbon with low polarity is typical adsorbents and is commonly used for removal of non- polar organic materials in water and wastewater treatment. The high porosity nature of the carbon provides a large surface area for contaminants to be adsorbed (Corapcioglu and Huang, 1987). The adsorption is result in the physical bond between the molecules. The molecules of the adsorbent attract other atoms/molecules at the interface. Hence, water contaminants are gathered at the surface. The attraction force between the adsorbents and adsorbates should be high to hinder the molecules dissolving in water solution. Some drawbacks of carbon adsorbents such as the high capacity of adsorption to specific adsorbates, black color of the carbon and the high cost of production led to grow the demand to search for alternative adsorbents to meet the needs. Natural plants contain cellulose,

hemicelluloses, lignin and protein (M. Bassyouni, 2012). These constituents are able to adsorb wide range of solutes, especially divalent metal cations (Aksu, 2002). Polyphenolic compounds, such as tannin and lignin which are found in the natural plants are considered to be the active sites to attract the heavy metal cations (Dakiky et al., 2002;Wang and Lin, 2010; Chantawong et al., 2003). Different types of Natural plants contents are used in the adsorption process such as coconut, rice straw, wheat straw and palm fibers (Mohan and Pittman, 2007). Coconut fibers have high lignin contents which increase the toughness, resilience and durability. Moreover fibers have high abrasion and rot (fungal and bacterial) resistance. Wheat straw contains cellulose, hemicelluloses, lignin, silica, and proteins. It contains lower silica content and higher lignin content than rice straw (Zvinowanda et al., 2009). Although the biomaterials have good advantages to be used as adsorbents, the extreme riation of pH and temperature have high influence on the biosorbent stability. Moreover their physical and morphological properties keep their applications limited specifically in packed columns. The physical and chemical resistance of the biosorbents can be improved by producing bio-composites (Alireza Kaboorani & Karl R. Englund, 2011). This will help for shaping and processing the biosrbents to be used in different applications.

Currently, there is no commercial bio-composites for water treatment. This can be mainly attributed to the low physical and chemical stability of the produced materials. (Babel and Kurniawan, 2004). The biosorbents are supported/reinforced by means of matrixes which are frequently of synthetic nature (organic or inorganic) such as polymers and minerals, or the biosorbents are crosslinked with itself, by adding adequate bonding agents. The most important aims of the study of the bio-composites is to maximize the bonding force between the matrix and dispersing phases (natural particles/fibers) at high adsorption capacity as much as possible. The bio-composites can be produced by different methods such as entrapment, ncapsulation, adhesion, crosslinking, anchorage or grafting techniques (Volesky, 2003).

Polyurethane (PU) consists of a chain of organic units joined by urethane (carbamate) links. Polyurethanes are formed through polyisocyanates and polyol molecules reaction through step-growth polymerization based on exothermic reaction. Polyurethane show advantages as: the availability, easy handling, versatility such as foam, malleable and resistant bio-composites. Polyurethane has high temperature, friction and UV radiation resistance (Shaidan et al., 2011 & M. Sh. Zoromba et al., 2012).

Due to the high reactivity of the isocyanate in polyurethane, It is possible to form strong unions with a partial or totally deacetylated chitin, specifically at the contact surface of both phases during polyurethane polymerization (Cestari et al., 2004). The amount of adsorbates that can be removed by an adsorbent is a function of the characteristics, concentration and temperature (Lee et al., 2007). The characteristics of the adsorbates include: solubility, polarity, hydrocarbon saturation molecular structure and weight. Generally, the amount of material absorbed is determined as a function of the concentration at a constant temperature, which called adsorption isotherm (Shan Ho, 2006). Adsorption isotherms are developed by exposing a given amount of adsorbates in a fixed volume of liquids to varying amount of adsorbents (Malek and Farooq, 2006). In order to investigate the adsorption isotherm, two equilibrium models, Langmuir and Freundlich, were analyzed. (Sekar et al., 2004).

2. Material and Methods
2.1. Raw materials
Polyol (castor oil) was purchased from A.S.O company, India . Water which is captured in natural oil was removed by heating the polyol at 105 ^{o}C for 4 h under vacuum. Polymeric diphenyl methane di-isocynate (PMDI) was obtained from Almotaheda Company, Egypt and used as received with NCO 33% wt/wt. Activated carbon was obtained from Samoral company for water and wastewater treatment, Egypt. Coconut coir fibers, palm fibers, rice and wheat straw were collected from the fields in Egypt. The collected natural fibres were ground in a ball-mill at 250 r.p.m. for 4 h.

2.2. Polyurethane synthesis
Distilled water was added to castor oil (4.2 % wt/wt) in presence of dispersing agent (Anfomu 5000). Polymeric di-phenyl di-isocynates (PMDI) was added to the polyol gradually at room temperature; cooling system was used to keep the temperature of the exothermic system around 30 ^{o}C. Mixing time is 20 minutes with 1000 r.p.m. speed.

2.3. Preparation of natural fibers
Natural fibers were collected, ground and then sieved to obtain an average grain size less than 200 μm. Surface preparations were carried out using steam as well as dilute sodium hydroxide 5 (w/w) % to improve the surface roughness. Ground natural fibers were dried at 105 oC for two days. The treated ground natural fibers were then thoroughly rinsed in distilled water several times to remove the excess of NaOH that could otherwise continue to degrade the ground natural fibers' cell wall components. The

treated ground fibers were dried immediately to equilibrium conditions in an air circulated oven at 105 ^{o}C for 24 hours before storage and use.

2.4 Bio-composites

Natural fibers were sieved to maintain particle size less than 200μm. Its weight fraction was 30%. Ground natural fibers were added to the polyurethane at the beginning of polymerization reaction with mixing rate 1000 r.p.m. The bio-composite solution with high viscosity was injected into the mould after 30 minutes of intensive stirring.

2.5 Mould

Aluminum mould with a Teflon layer coating was used to prepare the PU- ground natural fiber bio-composites. Figure1 shows the items of the mould. The bio-composite product shape is hollow sphere with outside and inside diameters: 30 mm and 25 mm respectively.

Figure 1. Mould for bio-composites processing

2.6 Microscopy measurements

The morphology and dimensions of the ground palm fibers and rice straw were tested using scanning electron microscopy.   Samples were fractured in liquid nitogrgen. Micolayers of gold were sprayed over the fractured surface to make it conductive. Fractured surfaces of the bio-composites were examined under a scanning electron microscope (SEM), JEOL model JXA 840A (ADS+OM-Japan).

2.7 Adsorption unit

To study the adsorption process of the synthesized bio-composite material; 1200 mm glass column with internal diameter 50 mm was manufactured. The column has valve every 100 mm to take samples. The column was packed with hollow spheres (30 mm diameter) of polyurethane biocompsites materials. The experimental set-up is illustrated in figure (2). Commercial activated carbon (Samoral company, Egypt) has been used in the assessment. Polypropylene feed tank was used with capacity of 10 liters. The fresh water from Nile River was collected and pumped into the packed column. Polyproylene tank with 20 L capacity was used for treated water collection. The adsorption process was carried out within contact time of 30 min at room

temperature. The TDS ions were determined at 520 nm and compared with standard and blank solutions (Dakiky, 2002). Short contact time was fixed and assigned for this work to study the possibility of applying the synthesized bio-composite adsorbents in the treatment of fresh water in industries. Treated water is supposed to be used in water cooling system. The efficiency of removal (E) of the adsorbent on TDS, is calculated by:

$$E (\%) = [(C_o - C_1) / C_o] \times 100$$ where C_1 and C_o are equilibrium TDS and initial concentration in fresh water (mg/l), respectively. The TDS sorption was investigated by varying the adsorbents concentrations from 1 to 10 g/l, at room temperature and constant pH = 7.3 through 30 min contact time.

2.8 Adsorption isotherm

Langmuir and Freundlich adsorption isotherm models are widely used for fitting the data to investigate the relationship between sorbed and aqueous concentration at equilibrium. The Langmuir model assumes that the monolayer adsorption of ions takes place on a homogenous surface with no interactions among the adsorbed ions. The Langmuir model can be presented by:

$$q_e = \frac{K_L C_e}{1 + a_L C_e}$$

where q_e (mg/g) is the amount of ions adsorbed onto the unit mass of the adsorbent. K_L is the Langmuir equilibrium constant; C_e is the concentration of ionin solution , and α_L is the Langmuir constant (Aksu, 2002). The constants K_L which indicates the reactivity of binding sites and α_L are the characteristics of the Langmuir equation and can be determined from a linearized form of Langmuir model.
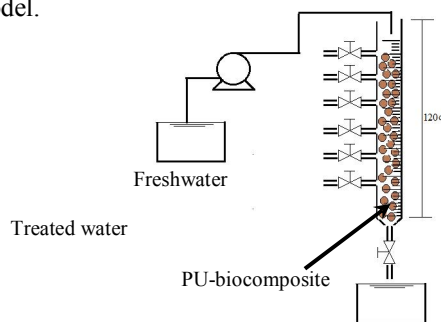

Figure 2. Adsorption unit

Langmuir isotherm can be linearized as four different types (Table 1), and simple linear regression will give different parameter estimates. The common linear forms used are Langmuir-1 and Langmuir-2. In this study, Langmuir-2 is applied.

Table 1. Isotherm parameters obtained using the non-linear method (Yuh-Shan Ho, 2006).

Isotherm		Linear form	Plot
Freundlich	$q_e = K_F C_e^{1/n}$	$\log(q_e) = \log(K_F) + 1/n\log(C_e)$	$\log(q_e)$ vs. $\log(C_e)$
Langmuir-1	$q_e = \dfrac{q_m K_a C_e}{1 + K_a C_e}$	$\dfrac{C_e}{q_e} = \dfrac{1}{q_m} C_e + \dfrac{1}{K_a q_m}$	$\dfrac{C_e}{q_e}$ vs. C_e
Langmuir-2		$\dfrac{1}{q_e} = \left(\dfrac{1}{K_a q_m}\right)\dfrac{1}{C_e} + \dfrac{1}{q_m}$	$\dfrac{1}{q_e}$ vs. $\dfrac{1}{C_e}$
Langmuir-3		$q_e = q_m - \left(\dfrac{1}{K_a}\right)\dfrac{q_e}{C_e}$	q_e vs. $\dfrac{q_e}{C_e}$
Langmuir-4		$\dfrac{q_e}{C_e} = K_a q_m - K_a q_e$	$\dfrac{q_e}{C_e}$ vs. q_e
Redlich-Peterson	$q_e = \dfrac{AC_e}{1 + BC_e^{g}}$	$\ln\left(A\dfrac{C_e}{q_e} - 1\right) = g\ln(C_e) + \ln(B)$	$\ln\left(A\dfrac{C_e}{q_e} - 1\right)$ vs. $\ln(C_e)$

Freundlich equation is an empirical equation based on adsorption on a heterogeneous surface. The equation is commonly given by:

$$q_e = aC_e^{b}$$

where a and b are the Freundlich constants. They indicate the adsorption capacity and the adsorption intensity, respectively. To simplify the derivation of a and b, above equation can be linearized as:

$$Log q_e = b \, log C_e + log a$$

Therefore, the constant a and exponent b can be determined by plotting log qe versus log Ce. The Langmuir and Freundlich models were applied to describe the values derived from the adsorption of TDS by each bioadsorbent through concentration range (1-10 g/l).

The plot of (1/Ce) versus (1/qe) using Langmiure-2 were established to investigate the fitting of the experimental results. Linear plots of log qe versus log Ce using Freundlich isotherm model was also used to represent the TDS adsorption by all types of adsorbent tested.

3. Results and discussion
3.1 Efficiency of TDS removal

The dependence of TDS sorption on PU-biocomposites was studied by varying the amount of adsorbents from 1 to 10 g/l. Figure 3 shows the TDS removal efficiency for four types of bio-composites adsorbents and the activated carbon at pH=7.3 and room temperature. Removal efficiency of the adsorbents is enhanced with increasing dose. This is can be attributed to the fact that the higher dose of adsorbents gives better probability of exchangeable sites for the ions in the water solution. Removal efficiency showed slight or no noticeable adsorption using activated carbon in 9-10g/lrange. It is supposed that after a particular concentration of adsorbent, the amount of ions attached and free remains constant even with increasing the concentration of adsorbent.

At 270 mg/l concentration of TDS, the maximum removal efficiency was found 37% for 10 grams of PU-ground palm fibers, while the maximum removal efficiency using PU-Ground rice straw, PU-Ground coconut coir and PU- Ground wheat straw were around 16%. The maximum removal using the activated carbon was 19.7% under the same operating conditions (Contact time, pH and Temperature). The high removal efficiency of palm fibers comparing to the other natural fibers can be attributed to the high contents of lignin (19% wt/wt) which has high adsorption capacity. Lignin contents in the other natural fibers are supposed to be around 6-10 wt/wt (Nabi Saheb et al, 1999).

Figure 3 shows the removal efficiency using the bicomposite adsorbents and activated carbon. It was observed that the removal efficiency of the bio-composite materials is higher than activated carbon at different doses.
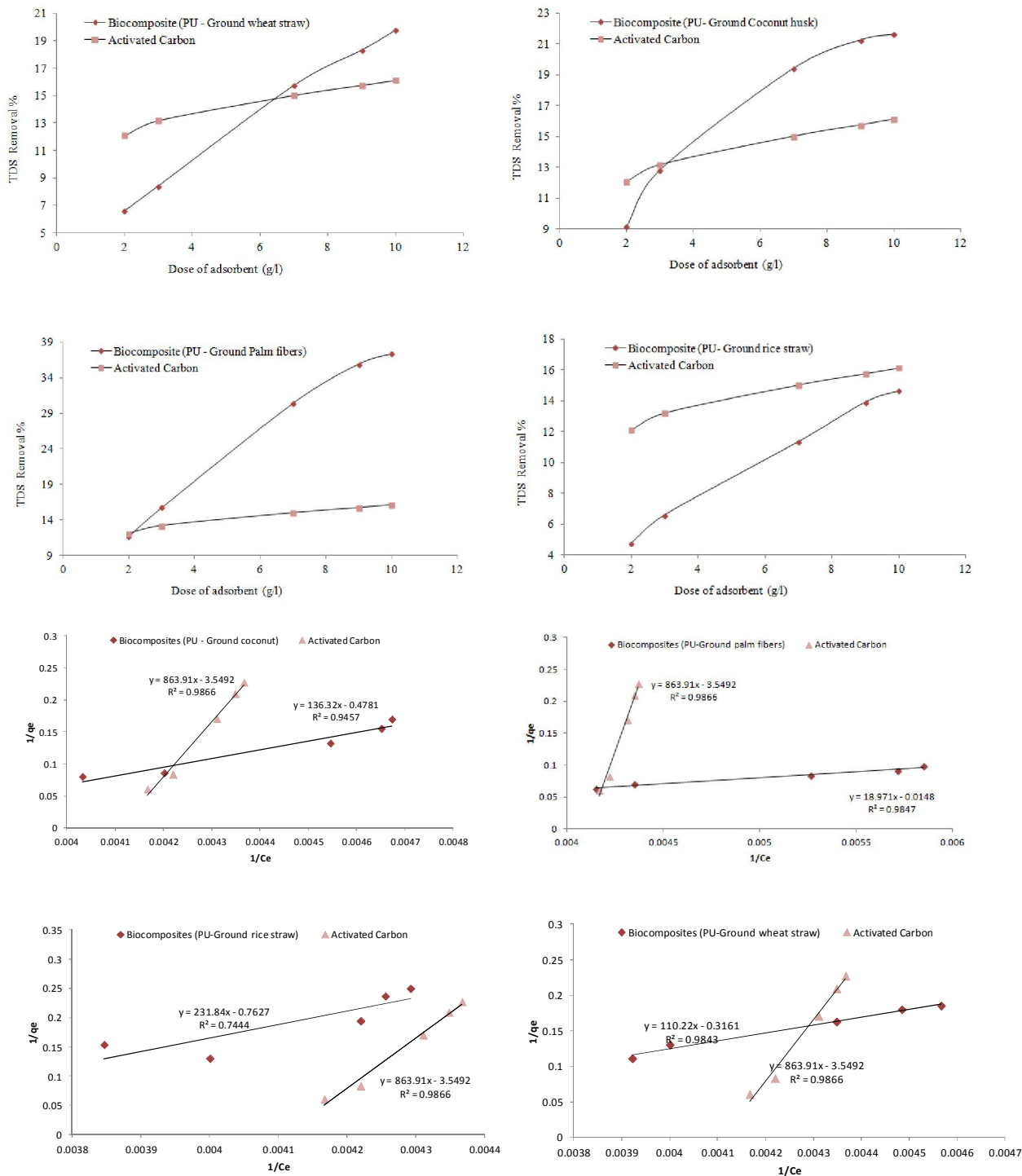
Figure 3. Effect of dose on the equilibrium removal efficiencies of
TDS using activated carbon and different bioadsorbents.

PU-ground coconut husk can be used to remove up to 22% of TDS using 10 g/l. PU- Ground wheat straw records a slight improvement above 6.3 g/l with respect to activated carbon. Ground rice straw shows low TDS removal comparing with commercial activated carbon. Significant improvement in TDS removal is obtained using PU-ground palm fiber.

It is noticed that the presence of cellulose and lignin (Polyphenolic compounds play a crucial role in the TDS removal. Rice straw has the lowest lignin content (14%) comparing with coconut (39 %), wheat straw (20 %) and palm fibers (25 %) (Amar K. Mohanty et.al., 2005). Cellulose represent around 48 % and 44% in palm leave and coconut respectively.

The ability of TDS removal using PU-biocomposites can be attributed to the presence of different adsorption materials capacities in bio-composites which can assist to collect and attract more TDS molecules.

3.2 Modeling and simulation of adsorption isotherm
In this study, Langumire and Freundlich models have been applied on biocomposites adsorbents involving ground (coconut husk, palm fibers, rice straw, wheat straw, and commercial activated carbon.

 Fig. 4 shows the fitting of the results using Langumire and Freundlich models. It is depicted that Freundlich model has a better data fitting than Langmuir as the bio-composite former have higher correlation regression coefficient than the latter.

This can be explained due to the fact that the foam cells and bioadsorbent materials formed a heterogeneous surfaces are used in colleting and attracting the TDS ions. On the other hand, the Langumire model has better fitting results for the activated carbon adsorbent indicating to formation of TDS monolayer on the surface of activated carbon adsorbent. This can be obtained when the adsorbent has a low surface area. Therefore, only monolayer of adsorbate is gathered on the surface of the adsorbent.
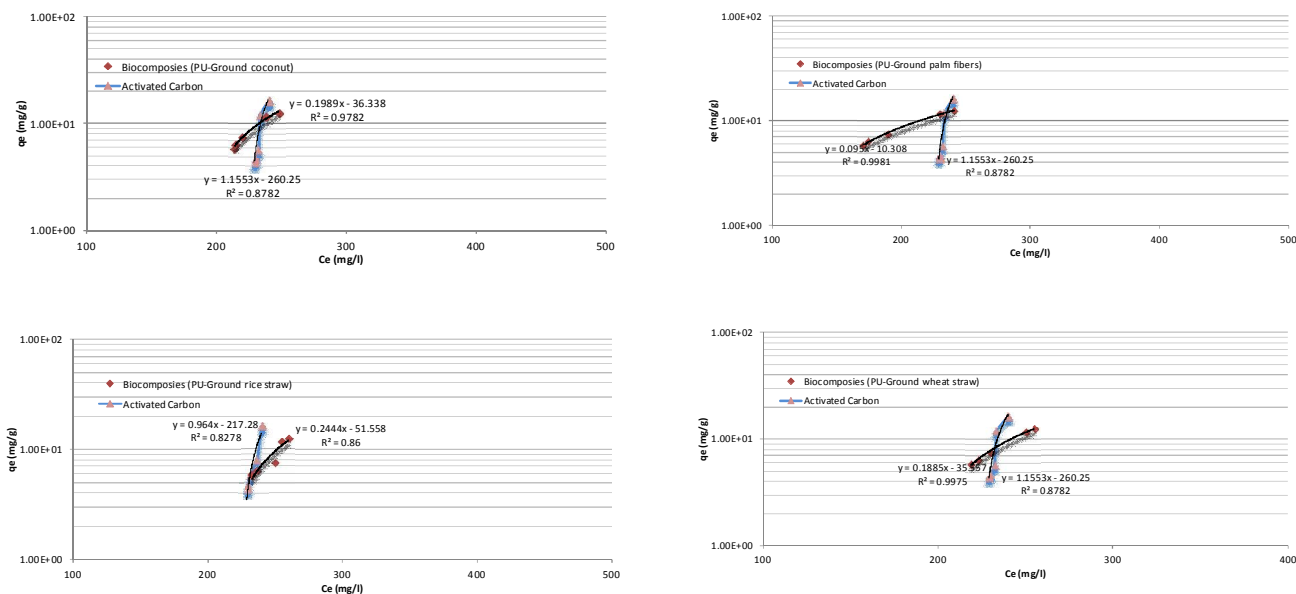

Figure 4. Isotherm models constants and correlation coefficients for adsorption of TDS from freshwater using various bio-composites adsorbents**.**

3.3 SEM analysis

Figure 5 shows the SEM surface analysis for the PU-biocomposites adsorbents. The foam cells and ground natural fibers including ground palm fibers (figure 5.a) and rice straw (figure 5.b) are dispersed well. It is believed that the rough surface of the ground natural fibers and the good distribution of the foam cells in the polymer assisted the biocomposite materials to adsorb the inorganic and organic substances contained in the fresh water and created strong interactions.
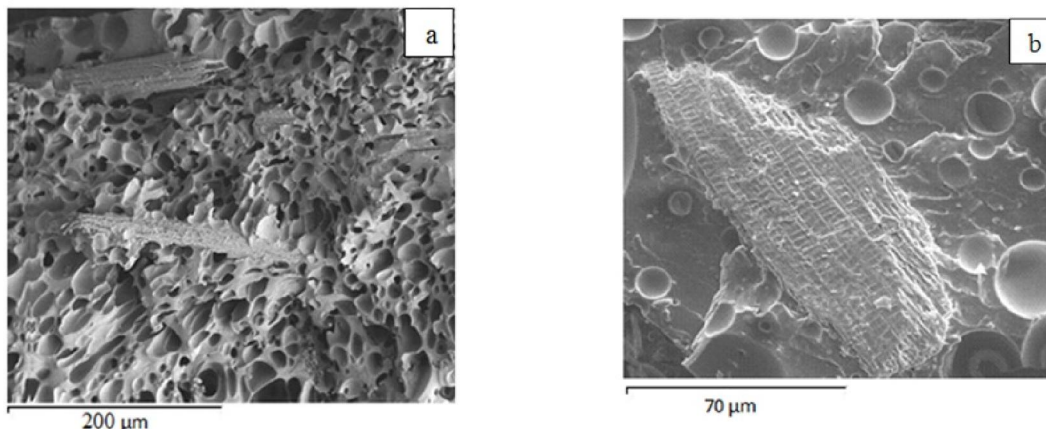

Fig.5 PU- ground natural fibers biocomposites, (a)
PU- Ground palm fibers. (b) PU-Ground rice straw

Conclusion

In conclusion, it has been shown that the use polyurethane biocomposites for total dissolved solids removal in fresh water appears to be applicable, eco-friendly and with high adsorption capacity. Moreover the foamed PU- ground palm fibers showed superior adsorption capacity properties. Using agricultural waste in water treatment will assist in reduction of waste generation.

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