

Nutrient removal using spent coconut husks

Trina Halfhide^{a,*}, Lorele J. Lalgee^b, Karen Seudat Singh^a, Joshua Williams^a,
Matthew Sealy^b, Anton Manoo^a and Azad Mohammed^a

^a Department of Life Sciences, Faculty of Science and Technology, University of the West Indies, St Augustine, Trinidad

^b Department of Chemistry, Faculty of Science and Technology, University of the West Indies, St Augustine, Trinidad

*Corresponding author. E-mail: trina.halfhide@sta.uwi.edu

Abstract

Wastewater management poses major environmental and economic problems in many small island developing states (SIDs), such as Trinidad and Tobago. Conventional wastewater treatment using activated carbon (AC) from charcoal is very costly. However, using spent coconut husks can be a cheap, environmentally friendly alternative source of AC for removing nitrogen and phosphorus in the secondary effluent. This study considered thermal and non-thermal, chemical activation methods to create four different AC treatments. The treatments considered included: virgin AC (thermally activated only) and subsequent acid treatments. Acid treatments were considered as an acidic environment, optimized nitrate and phosphate removal and reduced hydroxyl ion competition. Although all treatments effectively removed nitrates, virgin AC was ineffective in $\text{PO}_4^{3-}\text{-P}$ removal when concentrations were less than 10 mg/L. This was probably due to naturally occurring $\text{PO}_4^{3-}\text{-P}$ leaching from the coconut husk. The differences in removal efficiencies and adsorption capacities were not significantly different between treatments. Thermal activation was sufficient to remove $\text{NO}_3^- \text{-N}$ and $\text{PO}_4^{3-}\text{-P}$ greater than 10 mg/L in experiments with batch stock solutions and the secondary effluent. Virgin AC can therefore be used as a potential substitute for conventional AC.

Key words: activated carbon, adsorption, biochar, decentralized wastewater treatment options, secondary effluent, small island developing states (SIDs), water treatment

INTRODUCTION

Centralized wastewater treatment systems usually have high start-up and operational costs. Mechanical equipment requires for a large investment in start-up capital and then later requires constant maintenance. In addition, regular chemical inputs, such as sorbents and methanol, add to the operational costs. Most operational wastewater treatment plants (WWTPs) in small island developing states (SIDs), such as Trinidad and Tobago, utilize preliminary and primary sedimentation treatments. The effluent from these WWTPs is still nitrogen- and phosphorus-enriched. In recent years, more attention has been placed on water conservation, reuse and advanced treatment technology that can increase operational costs. Some of these costs can be circumvented by using cheaper alternatives such as agricultural waste products to make sorbents, such as activated carbon (AC). Spent coconut husks can be used to make AC that could be utilized in small decentralized wastewater treatment systems in SIDs. This approach seems logical when trying to tackle solid waste and wastewater problems in SIDs.

In Trinidad and Tobago, the median solid waste generated is approximately 1.8 kg per person per day, and this amounts to an estimated 1,000 tonnes per day (Pillai & Riverol 2018). Organic and food waste

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accounted for a third of solid waste in two major landfills in Trinidad (Singh *et al.* 2009). Most of this waste is misspent and becomes a source of unregulated methane production in the landfill sites. When specifically considering coconuts, Trinidad produces approximately 20,000 tonnes of coconut annually for local consumption (FAO 2018) and imports additional coconuts from Guyana. These husks are therefore a cheap and abundant agricultural waste in Trinidad and Tobago that can be used more appropriately as an adsorbent. The mesocarp of the coconut consists of 33–35% of husks (Tan *et al.* 2008). Its high carbon content comprises mainly of cellulose and lignin (60%). The presence of carboxylic, phenolic and hydroxyl groups makes them a useful adsorbent (Low *et al.* 1995; Al-Aoh *et al.* 2013).

Adsorption using AC is favored due to its high removal efficiencies with a wide range of pollutants, fast adsorption kinetics and its simplistic design (Tan *et al.* 2008). Using AC can be more affordable and environmentally friendly if sustainably sourced lignocellulosic agricultural waste is utilized (Cobb *et al.* 2012). Lignocellulosic agricultural wastes such as bamboo (Hameed *et al.* 2007), wood (Kilpimaa *et al.* 2015), rice husks (Cheenmatchaya & Kungwankunakorn 2014), banana and orange peels (Annadurai *et al.* 2002) and coconut shells (Gratiso *et al.* 2008) have all been used to prepare AC. Kilpimaa *et al.* (2015) demonstrated that AC derived from wood was suitable for phosphate and nitrate ($\text{PO}_4^{3-}\text{-P}$ and $\text{NO}_3^-\text{-N}$, respectively) removal and had a higher adsorption capacity than commercially available AC. Using lignocellulosic agricultural waste or by-products to make the plant-derived AC for wastewater treatment reduces the indirect environmental impacts associated with wastewater treatment, such as the reliance on nonrenewable materials such as coal for the production of AC. In addition, commercially producing AC from coal can be costly and does not allow for recycling water and nutrients (Cornejo 2015).

The production of AC from coconut husks requires either thermal or nonthermal activation. There are a number of factors that affect the AC produced: temperature, treatment time and chemical characteristics of husks. These factors influence the porosity, chemical impregnation ratio, surface characteristics and adsorption capacity of the AC (Tan *et al.* 2008). Thermal or physical activation increases pore development and surface area while decreasing the bulk mass (Kilpimaa *et al.* 2015). Laine *et al.* (1989) noted an optimal thermal activation temperature of 450 °C. Chemical activation is commonly used for materials that have high cellulosic content (Crittenden 2005). The main advantage of nonthermal chemical activation includes: lower activation temperatures, shorter activation times and high porosity development. A combination of thermal and nonthermal chemical treatments with coconut husks resulted in AC with higher removal efficiencies (Cobb *et al.* 2012).

The aim of this study was to investigate the effect of treatment methods on nitrate ($\text{NO}_3^-\text{-N}$) and phosphate ($\text{PO}_4^{3-}\text{-P}$) removal when considering AC derived from coconut husks. Both thermal and subsequent nonthermal chemical activation techniques were considered. A thermal or physical activation step was observed by using a muffle furnace at 450 °C to produce virgin AC for all test treatments. After the thermal activation step, chemical activation using three acids such as HNO_3 , HCl and H_2SO_4 was considered. Afkhami *et al.* (2007) and Kilpimaa *et al.* (2015) showed that acid treatment results in protonation of the adsorbent surface, increasing the positive surface charge density and enhancing the adsorption capacity towards negatively charged species, such as $\text{PO}_4^{3-}\text{-P}$ and $\text{NO}_3^-\text{-N}$ removal, as anions competed with hydroxyl (OH^-) groups at the AC surface under alkaline conditions. Batch adsorption experiments were conducted to determine the efficacy of physical and chemical activation treatment options.

METHOD

Preparation of activated carbon

The husk samples were from the tall variety of the species, *Coco nucifera*. Activation is required to transform the raw coconut husks into AC, which is effective in water treatment. Physical (thermal) and subsequent chemical (nonthermal) activation treatments were investigated.

Physical activation

Physical activation was achieved through a heat, combustion process called pyrolysis. Pyrolysis is a combustion process that allows for greater pore development and the reduction of bulk mass. Coconut husks were burned in a muffle furnace at 450 °C for 120 min to produce virgin AC (biochar).

Chemical activation

The virgin AC was further activated using three different acid treatments for 24 h. Acid treatments were chosen as to decrease repulsion forces when trying to remove PO₄³⁻-P and NO₃⁻-N anions. A mass of 3.0 g of virgin AC was measured, and 100 mL of 0.1 M of HCl, HNO₃ and H₂SO₄, respectively, was added for each treatment. After 24 h, the treated AC was rinsed with deionized water, filtered using a 0.45 µm pore glass fiber filter and then oven-dried at 60 °C for 24 h.

Experimental procedure

The experimental design comprised of batch equilibrium experiments to test the efficacy of nitrate and phosphate removal considering three individual feeds: (1) nitrate synthetic wastewater, (2) phosphate synthetic wastewater, and (3) wastewater from the fourth sedimentation tank in a biological treatment plant.

Batch equilibrium studies

Two equilibrium batch studies were conducted. The stock NO₃⁻-N solution was prepared from NaNO₃ and stock PO₄³⁻-P solution from Na₃PO₄. In the first equilibrium study, a volume of 25.0 mL with a concentration of 100 mg/L NO₃⁻-N (10.0 mg /L) and 150 mg/L and PO₄³⁻-P concentration (15.0 mg/L) was used. A mass of 0.03 g of each AC treatment was gently stirred manually at time 0 and then left latent for a 96-h interval. Nitrate MR, Cadmium Reduction Method 8171 in the HACH DR/2800 Spectrophotometer and Reactive (Orthophosphate) Method 8048 were conducted using the HACH Spectrophotometer (DR/2800) after the 96-h period.

In the second experiment, the secondary wastewater effluent from the fourth sedimentation tank in a low-technology biological WWTP was considered (refer to the section 'Batch equilibrium studies' below). An aliquot of 25.0 mL sample was used. The same NO₃⁻-N and PO₄³⁻-P spectrophotometer method from the first experiment was repeated.

Municipal wastewater feed

Approximately 10.0 L of wastewater from the fourth sedimentation pond was collected from a wastewater treatment facility in Piarcó, Trinidad. The daily flow in the facility is approximately 176 m³ per day (WASA 2012). The facility comprises the following: pretreatment (removal of large debris), four sedimentation tanks are set up in a series to reduce solids and nutrients, and followed by a final plug-flow reactor, where an unregimented chlorination process is conducted (Figure 1).

Adsorption capacity and nutrient removal efficiency

The amount of NO₃⁻-N and PO₄³⁻-P adsorbed was determined using Kilpimaa *et al.*'s (2015) equation:

$$q_e = \frac{(C_0 - C_e) V}{m} \quad (1)$$

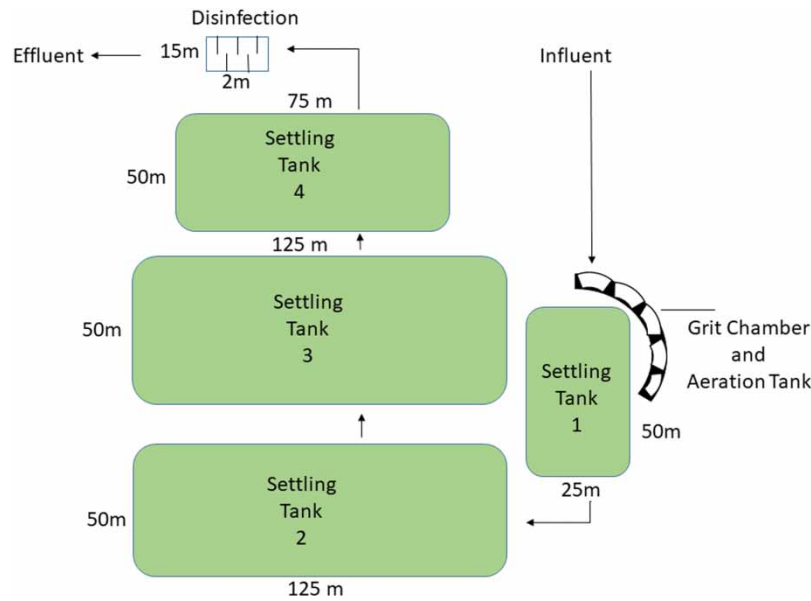


Figure 1

Figure 1 | Schematic of the WWTP where the secondary effluent was obtained.

where Q_e is the adsorption capacity, C_0 and C_e are the initial and equilibrium concentrations, V is the total volume (L) of the solution and m is the mass of the adsorbent (g).

Nutrient removal efficiency, determined as percentages, was calculated using the following equation:

$$\text{Overall removal efficiency (\%)} = \frac{C_i - C_f}{C_i} \times 100\% \quad (2)$$

where C_i is the initial nutrient concentration and C_f is the final nutrient concentration.

Adsorption models

Langmuir (Equation (3)) and Freundlich (Equation (4)) models were used to quantify the adsorption data and to gain a better understanding of the mechanism of adsorption, which depends on several factors such as the polarity (chemical functional groups present), surface area and porosity of the surface (Kołodziejńska *et al.* 2012). The Langmuir isotherm assumed a monolayer adsorption on a homogeneous surface, without interactions between the adsorbed species.

$$\frac{1}{q_e} = \frac{1}{q_m} + \frac{1}{q_m b C_e} \quad (3)$$

where Q_e (mg/g) is the amount of adsorbate adsorbed per unit mass of adsorbent, C_e (mg/L) is the concentration of the adsorbate at equilibrium, q_m (mg/g) represents the maximum adsorption capacity and b (L/mg) is a constant corresponding to the heat of adsorption and adsorption affinity of NO_3^- to binding sites on AC.

The Freundlich isotherm (Equation (4)) assumes that the adsorption process occurs on a heterogeneous surface.

$$\ln(q_e) = \ln(K_f) + \frac{1}{n} \ln(C_e) \quad (4)$$

where K_f is the Freundlich constant related to adsorption capacity (mg/g) and $1/n$ is the Freundlich constant related to the surface heterogeneity and the intensity of adsorption.

Surface characterization

Fourier transform infrared (FTIR) spectroscopy was used to determine the major functional groups present in the surface of the AC. The spectra were obtained at room temperature on the solid samples using the Perkin Elmer Spectrum 400 FTIR in the range of 4,000–600 cm^{-1} .

Scanning electron microscope (SEM) images were taken at 326 \times and 2,620 \times to observe surface physical characteristics with different AC treatments, with particular emphasis on apparent pores and cavities.

RESULTS AND DISCUSSION

Overall nutrient removal from equilibrium studies

NO_3^- -N and PO_4^{3-} -P removal from the stock solutions and wastewater were examined. Greater removal success was achieved with NO_3^- -N removal than PO_4^{3-} -P. NO_3^- -N removal in all treatments ranged between 74.0% and 90.3%, whereas PO_4^{3-} -P removal efficiencies had a greater range between -9.9% and 80.8% (Figure 2). Similar success was achieved when considering the NO_3^- -N removal in the WWTP secondary effluent, with a mean removal efficiency of 77.3% and a mean treated effluent concentration of 2.3 mg/L.

The AC treatments were also successful in removing PO_4^{3-} -P from the WWTP secondary effluent, with a mean removal efficiency of 57.9% and an effluent of 2.9 mg/L achieved.

When considering virgin AC (thermal activation only) with the low PO_4^{3-} -P test concentration (10.0 mg/L), there was a mean negative removal efficiency of 9.8% or an increase by approximately 1.0 mg/L PO_4^{3-} -P in the system. This was the only negative removal efficiency observed (Figure 2). This increase is due to the naturally occurring phosphorus associated with coconut husks leaching out and increasing PO_4^{3-} -P in the system. Torillo & Mihara (2012) estimated the total phosphorus found in coconut husks to be approximately 0.3037 mgP/g. Preliminary experiments with negative controls comprising of virgin AC and deionized water only also showed a similar increase of PO_4^{3-} -P over the 7-day experimental period (Figure 2(b)). Although this is the first study that notes PO_4^{3-} -P leaching from virgin AC derived from coconut husks, there are a few studies that documented leaching from biochars and AC. In Li *et al.*'s (2006) study, fly ash, a solid waste by-product from the coal-fired power plant, was considered for phosphate removal, but concerns arose as metals were leaching. In addition, Yao *et al.* (2012) showed varied leaching rates at different temperature regimes in peanut hull, sugarcane bagasse and bamboo biochar treatments. All bamboo treatments experienced leaching, regardless of the temperature regime used for pretreatment.

Adsorption capacity

Virgin AC (thermal activation only) and other treatments produced comparable NO_3^- -N removal efficiencies ranging between 74.0% and 90.3%. From the adsorption capacities calculated, AC with HNO_3 had the highest Q_e for both NO_3^- -N and PO_4^{3-} -P adsorption (Tables 1 and 2). The mean adsorption capacity (Q_e) for all treatments was similar, and mean differences between the four different treatments were not significant ($F = 2.19$; F -critical value = -3.49). Namasivayam & Sangeetha (2004) investigated the efficacy of ZnCl_2 -activated coir pith AC for the removal of PO_4^{3-} -P at various concentrations between 10.0 and 40.0 mg/L in aqueous solution. The mean adsorption capacity (Q_e) in Namasivayam & Sangeetha's (2004) study was similar to that seen in this study for virgin AC

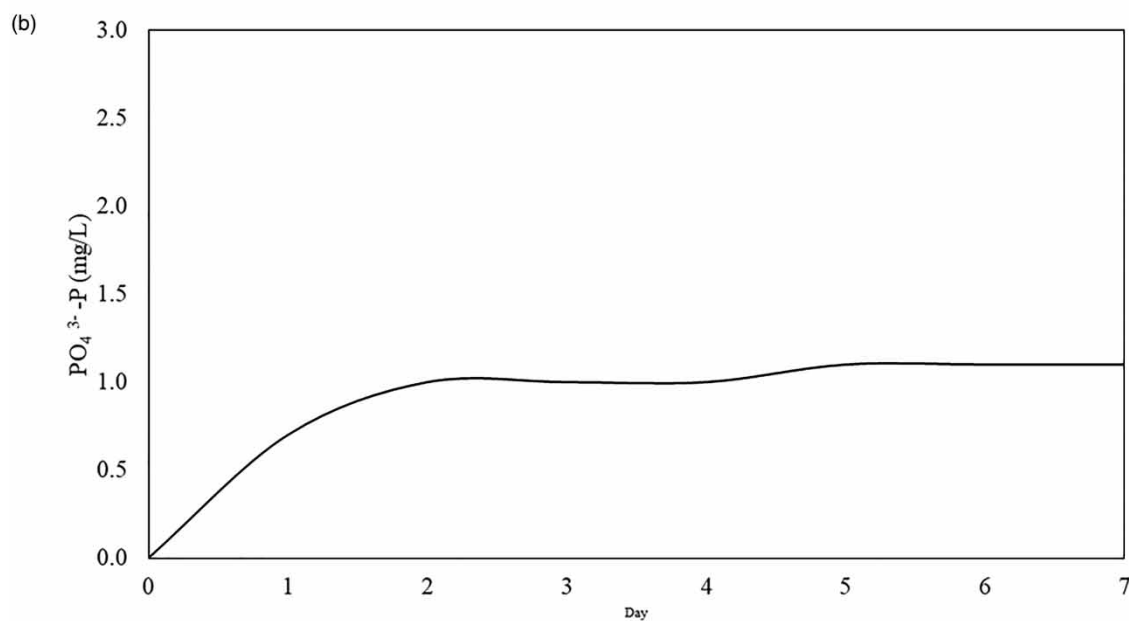
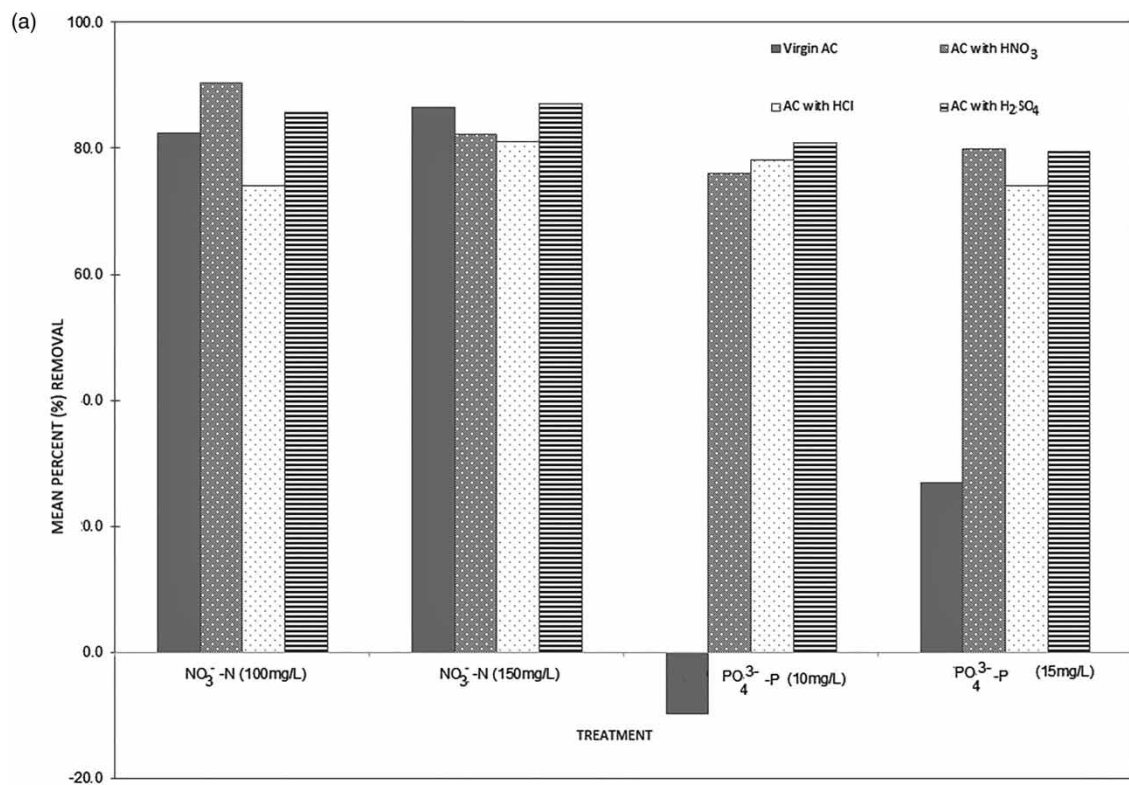


Figure 2 | (a) Mean removal efficiency for the four treatments. (b) Concentration of PO₄³⁻-P in the negative control with virgin AC.

Table 1 | Mean adsorption capacities of nitrates (NO₃⁻-N) considering the different AC pretreatments

Pretreatments	Q _e (mg/g)
Virgin AC	88.3
AC with HNO ₃	89.0
AC with HCl	81.5
AC with H ₂ SO ₄	88.4

Table 2 | Mean adsorption capacities of phosphates (PO₄³⁻-P) considering the different AC pretreatments

Pretreatments	Q _e (mg/g)
Virgin AC	1.3
AC with HNO ₃	8.2
AC with HCl	7.9
AC with H ₂ SO ₄	8.1

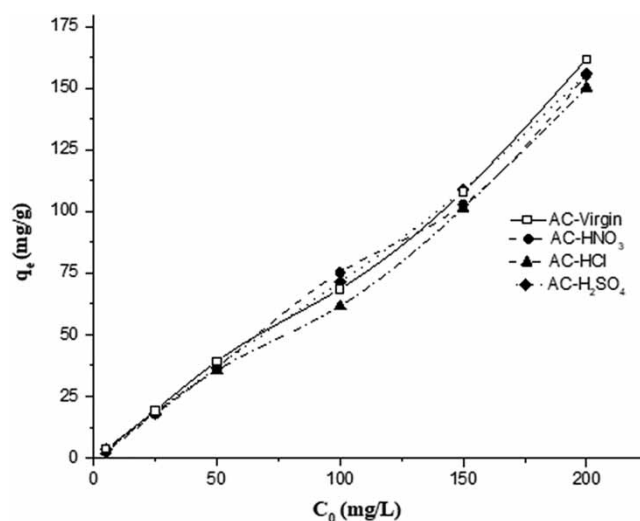
(1.3 mg/g). The observed Q_e was even greater for AC with acid treatments (7.9–8.2 mg/g). This suggests that acid treatments are more successful than a ZnCl₂ chemical activation treatment.

The adsorption affinity (b) and maximum adsorption capacity (q_m) decreased for the HCl-treated AC compared to the virgin AC (Figure 3). Increasing the NO₃⁻-N concentration increased the adsorption capacity of all the treatments (Figure 3), although the percentage removal varied at values greater than 50 mg/L NO₃⁻-N (Figure 4). Thermal and subsequent nonthermal activation processes were required to produce AC from raw coconut husks for the four treatments used in this study. This adsorption difference between AC treatments was not significant. Furthermore, the adsorption affinity (b) and maximum adsorption capacity (q_m) decreased for the HCl-treated AC. These results suggest that subsequent nonthermal, chemical activation was not necessary to increase NO₃⁻-N removal and that thermal activation was sufficient. Thermal activation increased the specific surface area and decreased the bulk mass significantly (Kilpimaa *et al.* 2015). Thermal activation to produce virgin biochar is advantageous for the environment as there are no chemicals utilized and less embodied energy involved in the treatment process.

The conventional Langmuir model was not valid for all AC samples and only fits the adsorption data for virgin AC and AC with HCl. The Freundlich model was applicable to all treatments with R^2 values ranging between 0.85 and 0.99 (Table 3), with the highest values obtained for AC with HNO₃ ($R^2 = 0.99$) and AC with H₂SO₄ samples ($R^2 = 0.92$). The adsorption isotherms of NO₃⁻-N showed the steepest slope for AC with HNO₃ and the least with virgin AC (Figure 5).

Understanding surface characteristics

Surface chemical composition plays an important role in the adsorption capacity of the AC. SEM images confirmed more physical similarities between virgin AC and AC with acid (HNO₃, HCl and

**Figure 3** | Initial concentration (C_0) vs. the adsorption capacity (q_e).

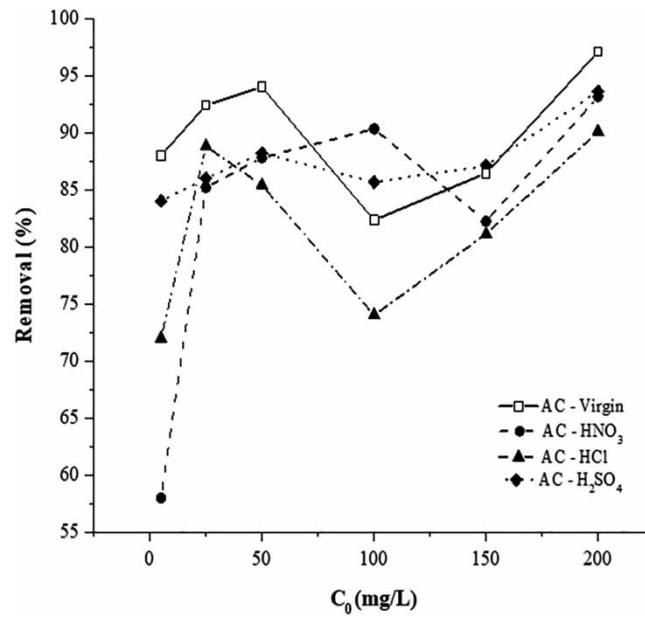


Figure 4 | Initial concentration (C₀) vs. the removal efficiency (%).

Table 3 | Freundlich model for all treatments

	K _f (L/g)	n	R ²
Virgin	16.05	1.72	0.85
HNO ₃	1.99	0.61	0.99
HCl	8.92	1.36	0.89
H ₂ SO ₄	4.88	0.90	0.92

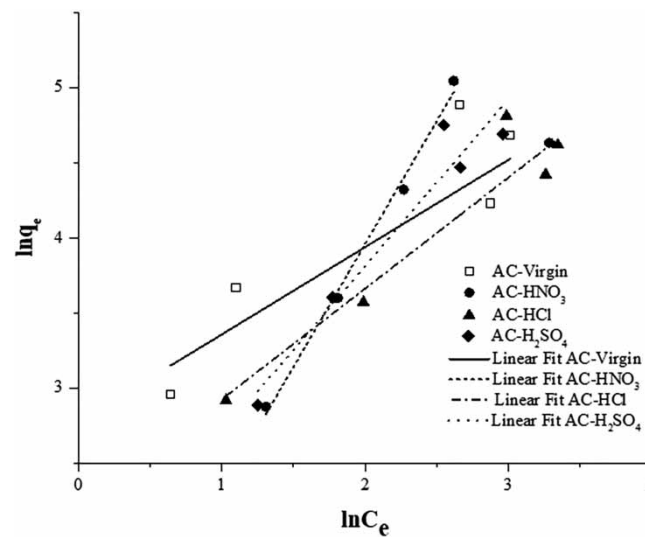


Figure 5 | Equilibrium concentration (C_e) vs. the adsorption capacity (q_e).

H₂SO₄) treatments (Figure 6). The highly varied AC surface observed with post-acid treatment could explain their similar adsorption characteristics and crystal structures at 2,620× magnification (Figure 6).

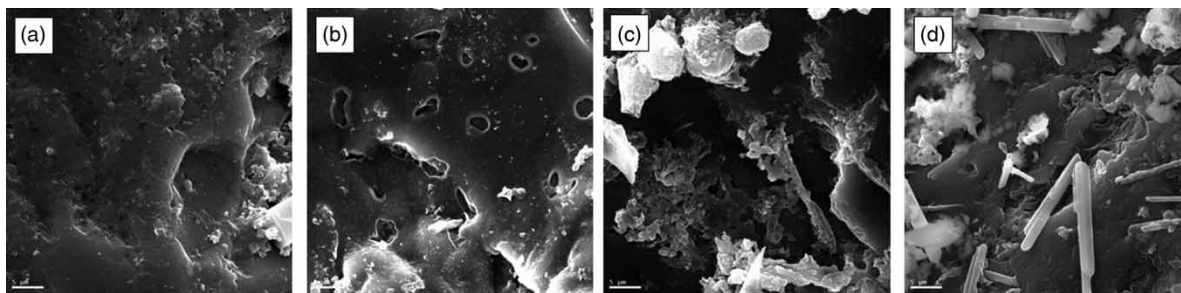


Figure 6 | (a) SEM image at 2,620 \times for virgin AC. (b) SEM image at 2,620 \times for HNO₃ treatment. (c) SEM image at 2,620 \times for HCl treatment. (d) SEM image at 2,620 \times for H₂SO₄ treatment.

The FTIR results showed qualitative differences between virgin AC and the three acid treatments (Figure 7). An unresolved shoulder in the spectrum of the virgin AC sample at 3,542.86 cm⁻¹, assigned to O–H stretching and physically absorbed water, was observed which overlapped with the band at 2,959.03 cm⁻¹. The virgin AC also displayed stretching vibrations at 1,726.48 and 1,662.99 cm⁻¹. Bands corresponding to aromatic C=C and C–C stretching vibration as well as C–H deformations were observed at 1,557, 1,468 and 1,423 cm⁻¹. The use of strong oxidizing agents, such as HNO₃ and H₂SO₄, modifies the surface oxygen functional groups, resulting in acidic groups on AC such as carboxylic acid and lactones. Acidic functional groups may result in a negative surface charge due to the release of protons from the carbon surface to the solution (Sharma & Upadhyay 2009; Ota *et al.* 2013). Consequently, there would be a repulsive force between negatively charged carbon surface and NO₃⁻ anions resulting in poor adsorption capacity. In further studies, the molarity of the acids should be varied as the concentration of H⁺ would influence adsorbent surface charge, ionization and speciation of the adsorbate.

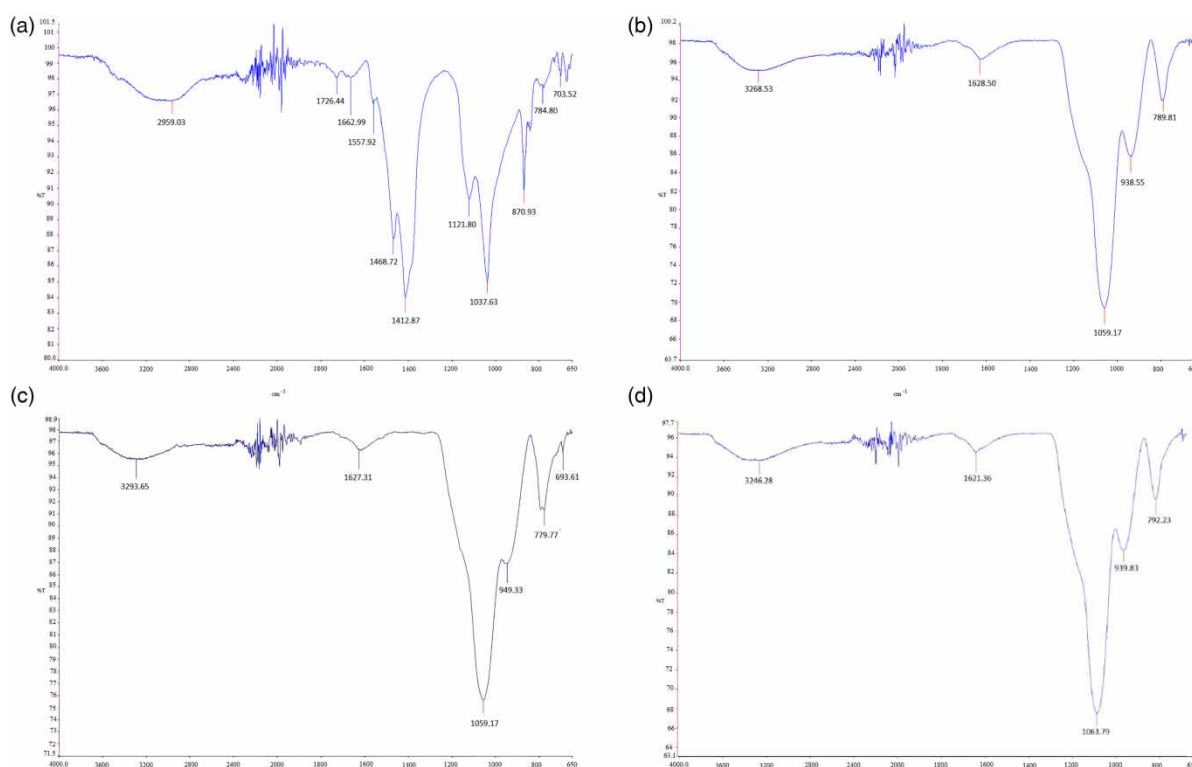


Figure 7 | (a) FTIR image at 2,620 \times for virgin AC. (b) FTIR image at 2,620 \times for HNO₃ treatment. (c) FTIR image at 2,620 \times for HCl treatment. (d) FTIR image at 2,620 \times for H₂SO₄ treatment.

In addition, acid treatment produced a greater number of surface functional groups. The spectra for each acid treated sample were similar but notable changes relative to virgin AC include: increasing in the intensity of the broad bands around 3,500–3,200 cm⁻¹ due to dehydration and alcohol oxidation which increases the concentration of carboxylic groups (-OH content); prominent bands at around 1,050–1,060 cm⁻¹ due to C–O stretching of alcohols, ethers, phenols and carbonyl systems arising from oxidation at the carbon surface due to acid treatment, as well as the introduction bands around 1,620–1,630 cm⁻¹ which can be assigned to C = C stretching vibrations of the substituted aromatics and C = O stretching of newly formed amide functionalities. Also, the absence of the bands in the region 1,560–1,400 cm⁻¹ may be a result of substitution on the aromatic ring. Although the acid treatments resulted in substantial structural changes, the physical changes were not significant to result in notable NO₃⁻ and PO₄³⁻ removal efficiencies. It should be noted that from preliminary experiments, most (mean = 73.3%) of NO₃⁻-N was adsorbed in the first 20 min for all treatments. Acidic functional groups may result in a negative surface charge due to the release of protons from the carbon surface to the solution (Sharma & Upadhyay 2009; Ota *et al.* 2013). Consequently, there would be a repulsive force between negatively charged carbon surface and NO₃⁻ anions resulting in poor adsorption capacity.

An unresolved shoulder in the spectrum of the virgin AC sample at 3,542.86 cm⁻¹, assigned to O–H stretching and physically adsorbed water, was observed which overlapped with the band at 2,959.03 cm⁻¹ attributed to the C–H group of long-chain aliphatic components (Chen & Wu 2004).

The virgin AC also displayed stretching vibrations at 1,726.48 and 1,662.99 cm⁻¹ attributed to C = O of C = O stretching of the ester functionality in hemi-cellulose and N–H bending in primary amines. Additional bands in the range of 1,120–1,030 cm⁻¹ indicate C–O stretching frequencies of functional groups such as alcohols, carboxylic acids and esters, consistent with the oxygenated functional groups of cellulose. Lower intensity bands 900–700 cm⁻¹ are due to the out-of-plane deformation mode of C–H in substituted benzene derivatives that most likely belong to lignin. The presence of hydroxyl groups, carbonyl group, ethers and aromatic compounds is consistent with the lignocellulosic structure of coconut husk.

Saman *et al.* (2015) noted that coconut husk generated AC that was homogenous and hence fitted well to the Langmuir adsorption model. This study utilized a higher activation temperature (700°C) and a base (KOH). Other research, including this study, showed a better fit with Freundlich. This suggests that there are multiple adsorption sites on the surface and the AC is heterogenous under these lower activation temperatures and acidic conditions. Junior *et al.* (2017) note that the pith is filled with many microstructures and interstices. These microstructures have different properties to conventional cellulose fibers, as they tend to have higher surface areas with hydroxyl groups.

Implications for industrial applications

AC adsorption proved to be successful for a wide range of pollutants (Tan *et al.* 2008). Although the selected treatment methods in this study did not make a significant difference in the adsorption capacity of NO₃⁻-N and PO₄³⁻-P concentrations greater than 10 mg/L, these may be more needed and useful for the removal of other substances such as hydrocarbons (Cheenmatchaya & Kungwan-kunakorn 2014), metals (Low *et al.* 1995; Annadurai *et al.* 2002; Jain *et al.* 2008) and other pollutants, such as dye (Al-Aoh *et al.* 2013). Generally, when considering cost and environmental impacts, it is better to have fewer processes. In particular, when considering cellulose nanocrystal extraction from the coconut fibers, using sulfuric acid in an industrial process increases environmental problems, such as terrestrial acidification. In addition, while one may consider increasing the concentration of sulfuric acid to improve removal efficiencies of AC, an increased concentration of sulfuric acid may speed the hydrolysis process, but the cellulose nanocrystal yield is reduced as the crystals' diameters are smaller. Using concentrated sulfuric acid in the AC and nanocrystal

production results in increased BOD, COD and total phosphorus emission loads (do Nascimento *et al.* 2016).

When PO₄³⁻-P concentrations are less than 50.0 mg/L, the effluent can be more difficult to treat and is referred to as advanced phosphorus or phosphate removal. Genz *et al.*'s (2004) study used simultaneous phosphorus removal processes, which include membrane bioreactors with enhanced biological removal and phosphorus precipitation, and results showed high PO₄³⁻-P concentrations between 100 and 300 mg/L. This study is one of the few that showed high Q_e success with the acid treatment of coconut AC.

Low maintenance and capital cost technologies have tremendous potential for decentralizing wastewater treatment and increasing nutrient removal. In addition to these nutrient removal benefits derived from AC production, there was a 96.6% reduction in mass and 99.0% reduction in volume. This makes this simple, low-cost technology even more attractive in SIDS and Caribbean states, such as Trinidad and Tobago, where space is always a valued asset and improper solid waste management presents economic, socio-political and environmental issues.

CONCLUSIONS

Using AC from spent coconut husks produces a useful sorbent. Adsorption was rapid as most of NO₃⁻-N was adsorbed in the first 20 min for all treatments. Using a thermal activation process to produce virgin AC is sufficient to reduce NO₃⁻-N, as there were no significant additional differences with subsequent chemical activation. Thermal and chemical activation treatments are necessary if PO₄³⁻-P concentrations are less than 10.0 mg/L. The results also indicated that surface chemistry more than structural properties governed the efficacy of the adsorption process.

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