

EVALUATION OF A GRAPHITE INTERCALATION COMPOUND FOR THE REMOVAL OF ORGANIC AND BIOLOGICAL CONTAMINANTS FROM GREY WATER SOLUTION BY ADSORPTION AND ELECTROCHEMICAL REGENERATION

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Abstract

Demand for clean water has increased considerably in recent years due to climate change and increased population density in urban and rural areas. Grey water recycling has become of increasing interest as a water conservation method in reducing stress on water resource. This report describes a novel approach to grey water treatment focusing on the removal of organic and biological contaminants. The process is based on the adsorption of contaminants onto a patented graphite intercalation compound supplied under the trade name of Nyex™. It utilises two fundamental elements: adsorption of contaminants and electrochemical regeneration of Nyex. Experimental results show that the uptake of organic contaminants onto Nyex is very fast with the majority of adsorption taking place within 5 minutes. A 4-log reduction in coliform was also observed with results from the electrochemical regeneration test indicating that up to 100% regeneration efficiency is achievable.

Keywords

Adsorption; Electrochemical regeneration; Graphite; Graphite intercalation compound (GIC); Grey water; Non-porous Carbon; Nyex™; Oxidation.

Introduction

Increase in population growth and extreme weather changes have been some of the contributing factors which has led to severe water shortage in many parts of the world. For this reason, several countries around the world have accepted and have embraced water reuse as a way of combating water shortage and draught (Jimenez and Asano 2008; Qadir et al. 2010; UNICEF and WHO 2012). The main methods by which water can be reused are through rainwater harvesting, grey water recycling and reuse of effluent from wastewater treatment plants. Of all these methods, grey water recycling is the most promising as it requires less costly pumping, its source is reliable and it has a low contaminant level (Pidou et al. 2007). Recycled grey water can be used untreated for non-contact use such as toilet flushing. However, there are health concerns if the untreated water is misused. In order to allow for other domestic and sanitation uses such as household cleaning and laundry, grey water can be processed through a number of treatment stages.

There are currently several manufactures of grey water recycling system, all trying to achieve the same goal of reducing the amount of mains portable water usage. Grey water is often processed through a combination of filtration, adsorption, chemical and biological treatment stages, similar to methods used for industrial wastewater treatment (Li et al. 2009; Pidou et al. 2007). Adsorption process represents a fundamental technology for wastewater treatment due to its capability to remove organic waste to extremely low level (McKay 1980). However, adsorption processes have gained very little attention in grey water recycling, and this is most likely due to the fact that the economic viability of the adsorption process greatly depends on the regeneration and reuse of the adsorbent.

The most common regeneration techniques are thermal regeneration, however, thermal regeneration is energy intensive which means high operating cost (Miguel et al. 2001). Other regeneration techniques which have also been used are wet air oxidation and chemical oxidation. Wet air oxidation requires high operating pressure, usually 0.5 – 20 MPa (Kolaczowski et al. 1999), thus limiting its application, whilst chemical regeneration has low regeneration efficiency and requires expensive and hazardous solvent (Berenguer et al. 2010). These drawbacks have encouraged research into alternative regeneration technique such as electrochemical regeneration. The electrochemical regeneration technique operates at mild atmospheric conditions (e.g. room temperature), has high regeneration efficiency, produces no waste and does not use hazardous chemicals ((Narbaiz and Karimi-Jashni 2009; Brown et al. 2004a,b). The operating cost of the electrochemical process depends on the conductivity of the adsorbent. Activated carbon is the most prolific adsorbent used in industry due to its high affinity to organic compounds (Halhouli et al. 1995). However, activated carbon adsorbent has moderate electrical conductivity (Narbaiz & Cen 1994).

Graphite intercalation compound (GIC) is graphite material treated with a strong reducing or strong oxidising agent, the formulation method of GIC provides it with high electrical conductivity (Fischer 1980; Chung 2002). Due to their high electrical conductivity, electrochemical activity and thermal insulation properties GIC has received vast interest for various uses. The insulating properties of GICs has led to their use as thermoelectric materials for thermal energy storage and in power sources such as electrodes and batteries (Inagaki 1989). GICs have only recently been investigated for adsorption purposes using a novel GIC adsorbent known as Nyex (Brown et al. 2004b, 2007). Nyex is a GIC with a high electric conductivity which results in quick regeneration and low operating voltage. The main aim of this report is to present findings from work undertaken to investigate if Nyex adsorbent is effective in removing organic and biological contaminants from grey water and to what extent.

Materials

Adsorbent (Nyex)

The Nyex adsorbent was supplied by Arvia™ Technology Ltd. It is a graphite intercalated compound (GIC) based on flake graphite with a basal (flat) surface which makes up the majority of the Nyex and an edge plane which is the corner of the flake. The non-porous nature of the adsorbent means it has a fairly high particle density of 2.2 g/cm³ with packed bed electrical conductivity of 0.8 S/cm. The particle size of Nyex was determined using sieve analysis. Nyex was found to have mean particle size diameter of 450 µm with particle size ranging from 100 to 1000 µm.

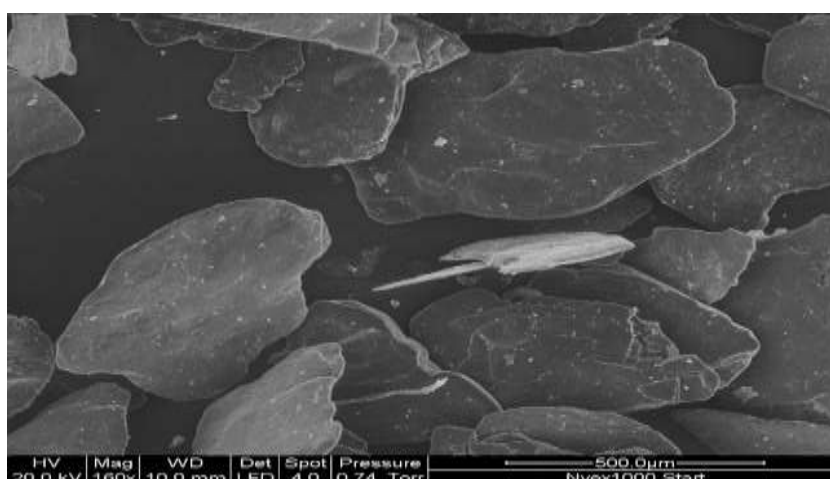


Figure 1: SEM micrograph of Nyex adsorbent used in this study (Mohammed et al.,2011)

Adsorbate (Grey water solution)

In order to reduce variability in the experimental conditions as well as to ensure reproducibility of data, synthetic grey water solution which mimicked grey water solution generated in the bathroom was formulated using guidelines provided by the British Standards (British Standards Institution 2011). The synthetic grey water was formulated using shower gel and sunflower oil to provide indicators for organic contaminants, septic effluent was also added to simulate biological as well as organic contaminants.

Experimental Details*Adsorption test*

For each set of experiment, 150 ml of synthetic grey water with known initial concentrations was mixed with a specific mass of adsorbent in a 250 ml Erlenmeyer flask. The flask was then shaken at a constant gentle speed of 260 rpm (Cole Parmer orbital shaker) enough to keep the adsorbent in suspension, and thus providing better mass transfer with high surface area of contact. Samples were collected at timed interval and were tested for organic contaminants by analysing the Chemical Oxygen Demand (COD) in mg/l.

The concentration of contaminant adsorbed (q) varies over time and was calculated as:

$$q(t) = \left(\frac{C_0 - C}{m} \right) V$$

Where C_0 is the initial concentration in mg/l, $C(t)$ is the concentration in mg/l at time t , m is the mass of the Nyex adsorbent used and V is the volume in ml of grey water solution.

The percentage reduction in contaminant was also used as an indicator to determine treatment efficiency and was estimated using the equation:

$$\% \text{ reduction} = \frac{C_0 - C}{m}$$

Electrochemical regeneration test

To test the regeneration efficiency of the system, a batch phase electrochemical regeneration study was carried out using a sequential batch reactor (SBR). The SBR consist of an anode and cathode compartment, separated by a micro-porous Deramic 350 membrane (Fig 1). The anode and cathode electrodes are both made of graphite material with an active area of 15 cm² for each electrode. The anode compartment holds the packed bed of adsorbent whilst

the cathode compartment consists of a catholyte solution made up of 3% NaCl solution. Method used for determining the regeneration efficiency was:

Step 1 - Initial adsorption: 50g of Nyex was added to the anode compartment along with 200 ml of synthetic grey water solution. Air is then pumped into the anode compartment to aid in mixing of the slurry. Samples collected were filtered and tested for COD contaminants.

Step 2 – Electrochemical regeneration: after the initial adsorption stage, the air flow is stopped and the Nyex adsorbent is left to settle into a packed bed, current is then passed across the cell to regenerate the bed of Nyex.

Step 3 – Re-adsorption: the content of the anode compartment was filtered leaving just the regenerated Nyex. 200 ml of synthetic grey water solution was again added to the anode compartment and adsorption was carried out using the same conditions as the initial adsorption step. The process was repeated over 5 cycles.

The percentage regeneration efficiency (RE) over the 5 cycles was estimated using the equation (Narbaiz and Cen 1994):

$$RE = \frac{q_r}{q_i} \times 100$$

Where q_i is the initial adsorption capacity of the fresh Nyex adsorbent obtained from cycle 1 and q_r is the adsorption capacity of the regenerated Nyex.

Continuous adsorption and regeneration test

Continuous adsorption and regeneration was achieved using an electrochemical cell separated into anode and cathode compartments (Figure 2). Both the anode and cathode are made up of graphite material with an active area of 625 cm². The anode compartment of the electrochemical cell was packed with enough adsorbent to cover the electrode and the cathode compartment contained the catholyte solution. The system was operated in a continuous mode where adsorption and regeneration occurs simultaneously. Meaning while the grey water was flowing through the adsorbent bed a DC current was passed across the cell.

During operation, grey water solution was pumped through a nozzle at the bottom of the anode compartment of the cell and allowed to flow through the packed bed. Grey water solution with initial concentration of around 724 mg/l was pumped through the packed bed of adsorbent at a known flowrate. Samples were collected from the top of the cell and analysed for COD and biological contaminants (E.coli).

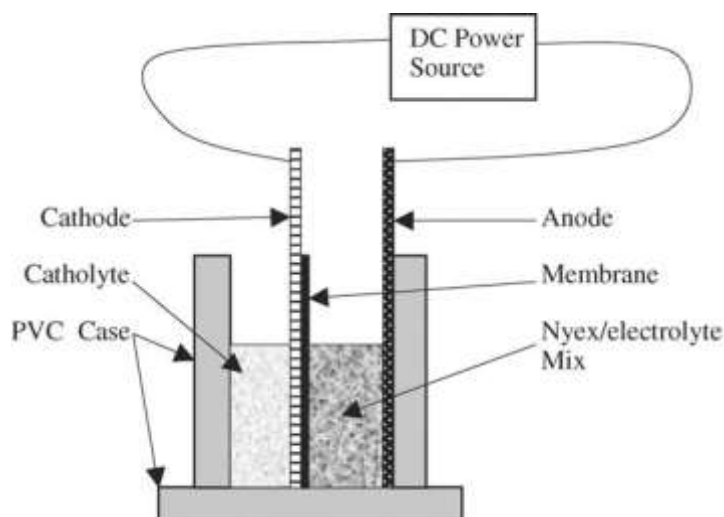


Figure 2: Schematic diagram of the electrochemical cell

Analysis

The COD was determined using the Hach-Lange photometric cuvette test. Sample was first added to the appropriate cuvette and then treated according to the manufactures manual. The treated cuvette is subsequently placed in a Hach-Lange DR 2800 spectrometer where the absorbance of the sample was measured at wavelength of 605nm. Biological analysis were undertaken by United Utilities Laboratory, using membrane filtration technique with membrane lactose glucuronide agar incubated at 44°C.

Results and Discussions

Adsorption kinetics

Adsorption kinetics was studied at room temperature with adsorbent concentration of 7g/l. The variations in the amount of organic contaminants adsorbed were noted in the series of contact time studied, and the result is presented in Figure 3. It can be seen from the plot that adding Nyex adsorbent to the grey water solution enabled removal of organics contaminants as indicated by the drop in bulk solution concentration with an increase in Nyex concentration from 0 to 7 g/l.

Adsorption of organics onto the Nyex adsorbent appear to occur in stages. The first stage of the curve is the steeper portion where Initially, with a relatively bare surface, the amount of available surface area for adsorption was high and consequently, the initial rate of adsorption was very high. The next stage is a rather constant portion with little changes in the amount of COD adsorbed due to a rapid decrease in adsorption site. This reduction in the fraction of active adsorption sites result in a slower adsorption rate over time as the system approaches

equilibrium. It is evident from the curve that the majority of the active sites available for adsorption were occupied in less than 5 minutes, which confirms that the adsorption of organic contaminants onto Nyex adsorbent is a fairly quick process. Rapid uptake of organic contaminants by Nyex have also been observed by Mohammed et al. (2011) and Brown et al. (2004b) in which around 80% of the active adsorption site was used up within the first 5 minutes. On the other hand, a study on adsorption onto various type of activated carbon showed that the majority of adsorption occurred within 40 minutes (Kannan and Sundaram 2001). The fast uptake of contaminants by Nyex adsorbent is attributed to the non-porous nature of the adsorbent, meaning pore diffusion is eliminated.

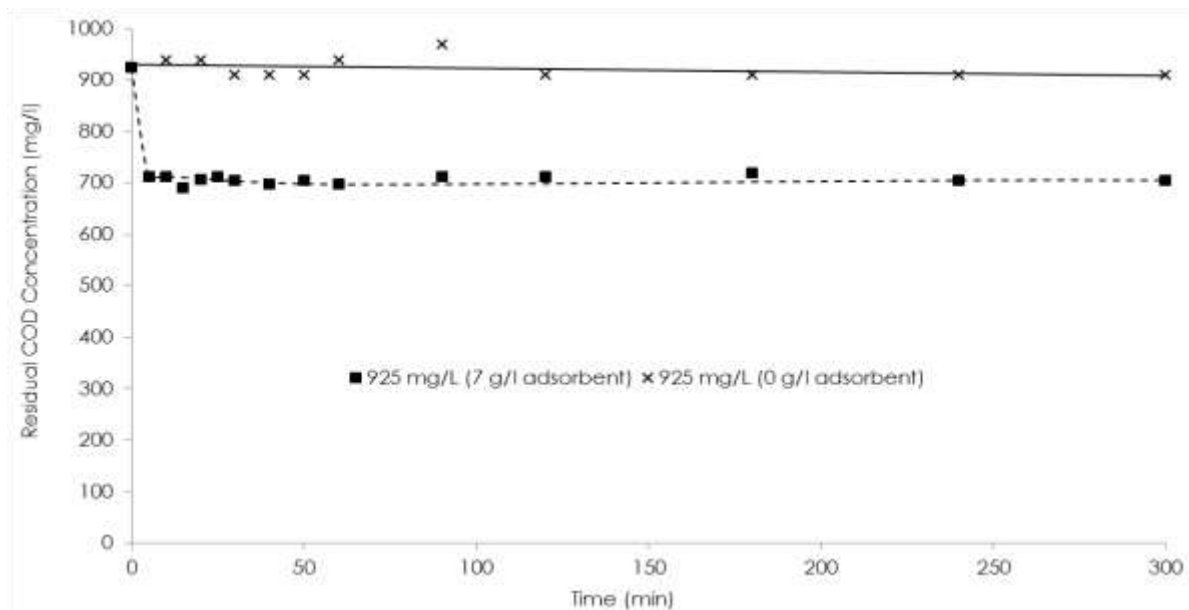


Figure 3: *Plot showing adsorption of organics (COD) with 0g/l and 7 g/l Nyex adsorbent, using 150 ml synthetic grey water solution with initial COD concentration of 925±50 mg/l*

In order to determine the rate limiting step of the adsorption, kinetic models were fitted to the experimental data. The pseudo-first order and pseudo-second order kinetic models were applied to evaluate the batch experiment data using a non-linear regression fit. The pseudo-first and pseudo-second order equation used to fit the experimental data are:

Pseudo-first-order rate equation:

$$\frac{dq}{dt} = k_1(q_e - q_t)$$

Pseudo-second-order rate equation:

$$\frac{dq}{dt} = k_2(q_e - q_t)^2$$

Where q_e and q_t are the adsorption capacity in mg/g of adsorbent at equilibrium and at time t , respectively and k_1 (1/min) and k_2 (g mg⁻¹ min⁻¹) are the first-order and second-order adsorption rate constant respectively.

The correlation coefficient value R^2 was used to determine the goodness of fit. The estimated adsorption capacity at equilibrium for the pseudo-first order kinetic model was obtained by trial and error in order to acquire the highest correlation coefficient value.

Results illustrated in Figure 4 indicates that the pseudo-first order kinetic model does not correlate very well with the experimental data over the range of time. This suggests that the pseudo-first order model is not a good representation of the adsorption process. On the other hand, good correlation was observed with the pseudo-second order kinetic model with a non-linear regression correlation coefficient value R^2 of over 0.96 compared to an R^2 value of 0.51 for the pseudo-first order kinetics.

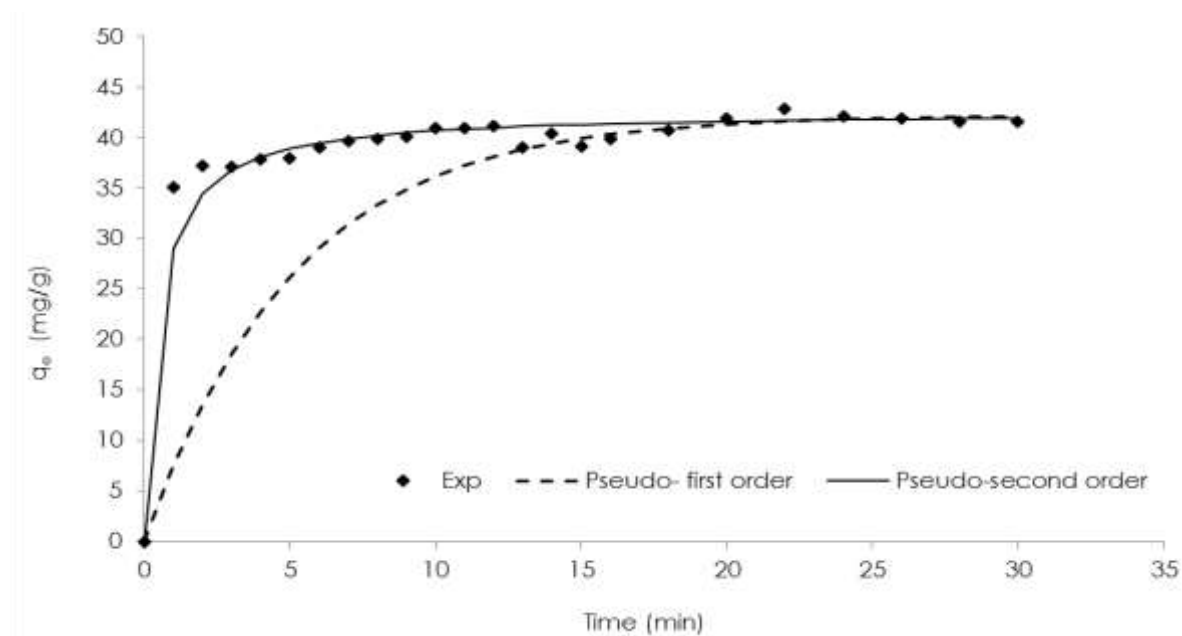


Figure 4: Adsorption kinetics: non-linear pseudo-first order and pseudo-second order kinetic fit to experimental data with initial COD concentration of 925±50 mg/l using 7 g/l Nyex adsorbent and 150 ml of grey water

A second order fit means that the adsorption rate of the system is proportional to the concentration squared. This supports the shape of the kinetics curve observed in Figure 5 where above an initial COD concentration of 480 mg/l, the equilibrium time increased from 5 minutes to around 15 minutes initially as depicted by the reduced steepness of the curve. The pseudo-second order fit of the kinetic experiment data also indicate the rate limiting step of the adsorption process is the chemical adsorption step (Ho and McKay 1998). A pseudo-second order fit was also reported for adsorption of organic contaminants from wastewater onto Nyex (Mohammed et al. 2011).

One of the concerns highlighted from using several different grey water treatment processes is the variability of influent grey water concentration. This has been found to have detrimental effects on the treatment efficiency especially on the biological treatment processes (Ward, 2000); Šostar-Turk et al. 2005; Lin et al. 2005; Eriksson et al. 2009). For this reason the effect of organic contaminant loading on treatment efficiency was investigated over a period of time. Figure 5 depicts the adsorption kinetics over a five hour contact time and at room temperature. The fast initial removal rate experienced at high initial organic concentration indicates that concentration difference is the primary driving force for adsorption of organic contaminants. Figure 6 reveal that the amount of organic contaminant removed from the bulk grey water solution was affected by the initial grey water concentration. An increase in removal efficiency is also indicated as the initial COD concentration increased from 88 to 925 mg/l. This supports the assumption of intermolecular attraction of the organic molecules possibly resulting in multilayer arrangement of organic molecules on the surface of the Nyex adsorbent (Boyd et al. 1947). In order to investigate the arrangement of organic contaminants on Nyex, an adsorption isotherm study was carried out.

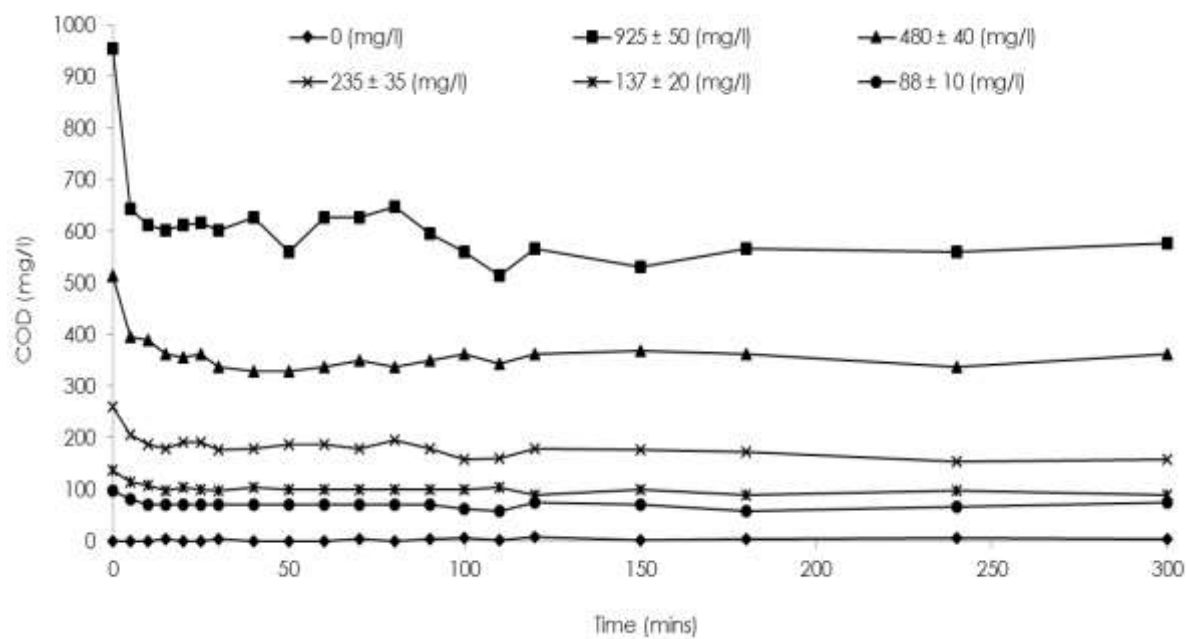


Figure 5: Effect of contact time on uptake of organic contaminants by Nyex adsorbent at various initial grey water concentrations, experiment carried out in batch mode using 16 g/l Nyex adsorbent and 150 ml of grey water

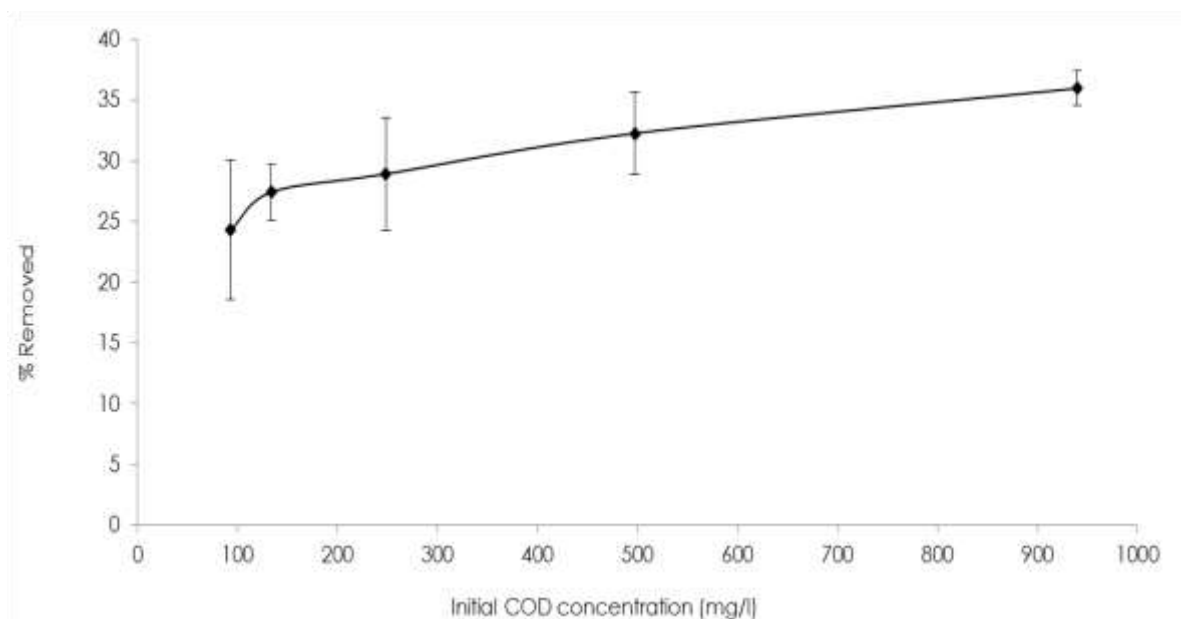


Figure 6: *Effect of initial grey water concentration on percentage removal of organic contaminants (COD), experiment was carried out in batch mode using 16 g/l Nyex adsorbent and 150 ml of grey water*

Adsorption isotherm

Based on the adsorption kinetic data, it was assumed that the system reaches equilibrium within around 30 minutes. Adsorption isotherm study was therefore conducted by mixing 7g/l Nyex adsorbent with a 150 ml grey water solution for 1 hour (to ensure equilibrium) at a range of concentrations between 0 and 925 mg/l.

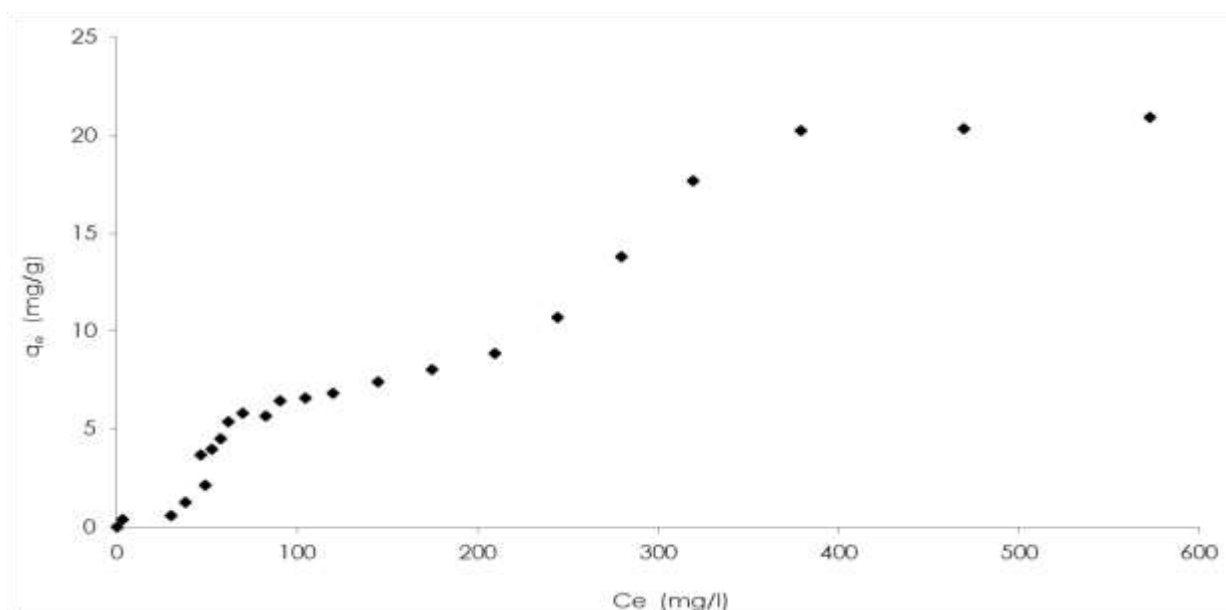


Figure 7: *Adsorption Isotherm result for organic COD removal carried over a contact time of 1 hour with 7 g/l adsorbent and 150 ml synthetic grey water solution*

The adsorption curve depicted in Figure 7 represents a Type 2 isotherm for multilayer adsorption (Brunauer *et al.* 1940). The adsorption isotherm shows a steep increase in solid phase equilibrium concentration (q_e) at COD concentrations below 100 mg/l until the majority/all of the active adsorption site had been occupied. This corresponds to a monolayer adsorption in which the contaminants bind strongly to the surface of the adsorbent. At grey water concentrations above 100 mg/l, no further adsorption took place after the entire active adsorption site was used up. This resulted in an increase in the liquid phase equilibrium concentration (C_e) with no further changes to q_e . There was another rapid increase in q_e at grey water concentration above 230 mg/l which suggests multilayer adsorption of contaminants at high grey water concentration. Multilayer adsorption occurs as a result of adsorbed organic molecules attracting other organic molecules from the bulk liquid phase. This leads to self-association of organic molecules on the Nyex due to intermolecular attraction, held together by Van der Waals force. The fact that a higher concentration is required in order to achieve multilayer adsorption implies that a high concentration gradient between the adsorbed and bulk liquid phase is required for stronger intermolecular attraction. This corresponds very well with previous results discussed earlier in Figure 5 and Figure 6.

Effects of Nyex adsorbent dose

To determine the effects of Nyex adsorbent concentration on the uptake of organic contaminant, batch experiments were performed using different mass of adsorbent.

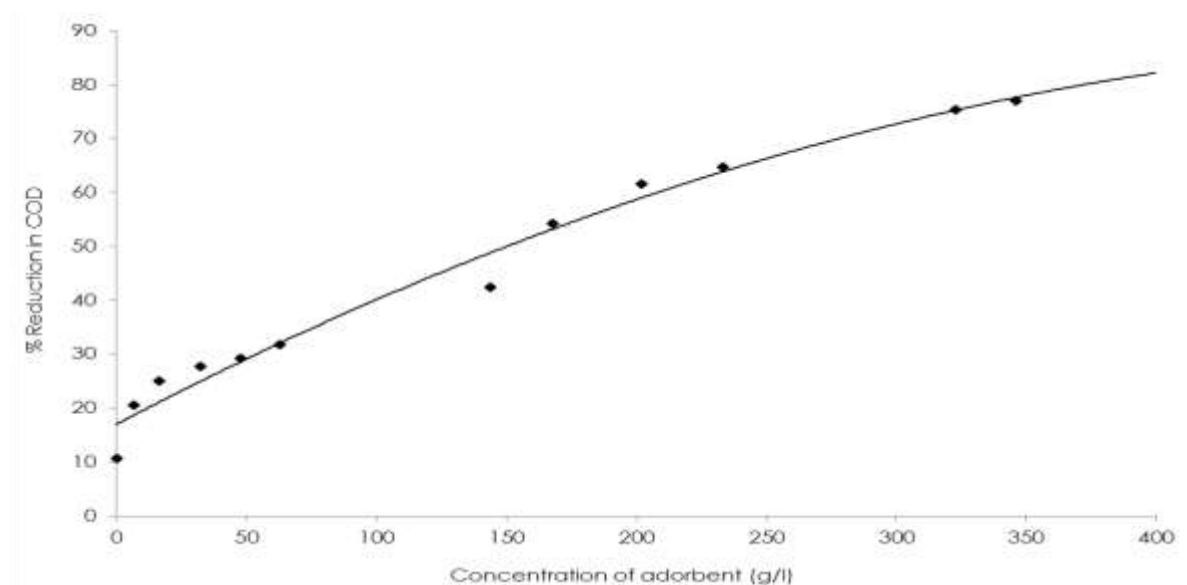


Figure 8: Effect of adsorbent mass on the reduction of organic contaminants (COD): batch experiment was performed with initial COD concentration of 724 ± 50 mg/l over a contact time of 1 hour

Due to increased adsorption surface area brought about by larger Nyex loading, it was expected that the uptake of contaminants by Nyex should increase as the Nyex dosing increase. As expected, the plot in Figure 8 shows that increasing the concentration of adsorbent increased the amount of organic contaminants adsorbed. What is surprising is that at adsorbent concentrations above approximately 150 g/l the curve starts to concave towards the concentration axis as a result of reduction in percentage removal with increase in Nyex adsorbent dosage. Similar results have been observed by Brown and Roberts (2007) for the adsorption of organics from solution using Nyex adsorbent. The decrease in percentage removed with increase in Nyex adsorbent concentration could be due to a combination of factors such as:

- Reduction in bulk phase COD concentration compared to the adsorbed phase concentration which results in a shift in the concentration gradient, and thus the adsorption driving force.
- Over lapping or coagulation of the Nyex adsorbent particles at high concentration resulting in a reduction in the amount of available adsorption sites. It has been observed that the Nyex adsorbent overlap on the basal plane when closely packed together thus leaving mainly the edge plane available for adsorption. Therefore indicating that a high proportion of organic contaminants adsorb on the basal plane.

The effect of coagulation or over lapping at high adsorbent concentration can be reduced by increasing mixing of the slurry. The effect of agitation speed was investigated by Aroua et al. 2008 on the kinetics of lead(II) adsorption onto palm shell-based activated carbon. The Author concluded that increasing the agitation speed result in an increased adsorption capacity due to improved distribution of the adsorbate in the bulk liquid phase and the reduction in the film boundary layer surrounding the adsorbent.

Regeneration efficiency

Data presented in this section reviews the use of electrochemical regeneration techniques in the regeneration of Nyex adsorbent saturated with organic contaminants adsorbed from grey water solution. The regeneration process involves desorption and/or destruction of the adsorbed organic contaminants by passing a current density of 20 mA/cm² across the loaded bed of adsorbent over a recorded period of time. The operating cell voltage during regeneration was 7.5 V.

The graph in Figure 9 shows that a 100% regeneration efficiency can be achieved with a charge of 2700 C which corresponds to a charge of around 187 C/mg COD removed. Passing no current across the cell would mean the Nyex adsorbent is saturated after the first cycle hence there should be zero adsorption after the first cycle. However, the plot indicates some

level of regeneration where adsorption was noted for each progressive cycle. The additional capacity noted for each cycle can be explained by the adsorption isotherm curve, where additional adsorption was noted through multilayer arrangement once the bulk phase concentration is increased over a certain point. In other words, the additional adsorption onto the unregenerate Nyex adsorbent is due to organic contaminants in the adsorbed phase attracting other molecules in the bulk liquid phase thus resulting in multilayer arrangement held together by Van der Waals force.

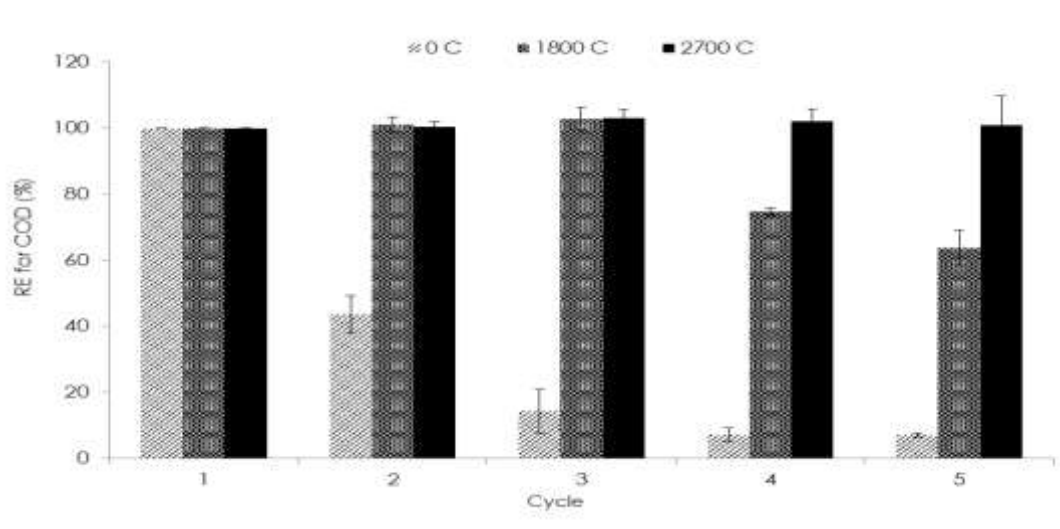


Figure 9: Regeneration efficiency data to study the regeneration efficiency in batch mode for COD contaminants 50 g of Nyex adsorbent was used with an initial COD concentration of 200 ± 20 mg/l and 3 g/l NaCl

Removal of biological contaminants by continuous adsorption/regeneration

The removal of biological contaminants from synthetic grey water solution was investigated by operating the system in continuous adsorption and regeneration mode as described in the experiment section.

Table 1: Operation test: effect of running the electrochemical cell for 6 hours at feed flow rate of 10 l/h, current density of 14.5 mA/cm² and at different initial biological loading on the removal efficiency of biological contaminants

Time (min)	E. coli (cfu/100 ml)	Pseudomonas (cfu/100 ml)	Time (min)	E. coli (cfu/100 ml)	Pseudomonas (cfu/100 ml)
0	6300	80000	0	610000	190000
60	<10	<10	60	<10	<10
120	<10	<10	120	<10	<10
180	<10	<10	180	<10	<10
240	<10	<10	240	<10	<10
300	<10	<10	300	<10	<10
360	<10	<10	360	<10	<10

Table 1 shows that operating the system in continuous adsorption and regeneration mode resulted in up to 4-log continuous reduction of biological contaminants for 6 hours. The continuous high removal efficiency noted is most likely due to the production of choline in the electrochemical cell which helped in disinfecting the grey water.

Conclusions

The characteristics of Nyex adsorbent for the removal of organic contaminants from grey water solution was investigated in this study. Batch experiments were carried out by mixing known concentration of synthetic grey water with a selected dose of Nyex adsorbent at various operating conditions. In order to determine the mechanism of adsorption and the equilibrium characteristics, the experimental results were fitted to well-developed adsorption kinetic models. The effect of initial grey water concentration and adsorbent dosage were also investigated.

1. Results showed that the initial uptake rate of organic contaminants by Nyex is very high with the majority of the active adsorption site being occupied in less than 5 minutes at high initial grey water concentrations of over 480 mg/l. This is most likely due to the non-porous nature of the Nyex which eliminate the intra-particle/pore diffusion step of the adsorption process. The pseudo-second order adsorption kinetic model provided the best fit for the adsorption kinetic data which denotes that adsorption of organics onto Nyex is by bond formation between the organic contaminants and functional groups on the Nyex, known as chemical adsorption. It was also observed that the uptake rate and efficiency of organic contaminants by Nyex increased with increasing initial grey water concentration. The high removal efficiency and fast initial uptake rate is as a result of high concentration driving force and increase in intra-particle attraction brought about as a result of high organic contaminant loading.
2. The adsorption isotherm curve illustrates multi-layer adsorption of organic contaminants onto Nyex, with strong chemical adsorption on the surface of the Nyex and then subsequent layers are formed via Van der Waals bond formation.
3. The effect of Nyex dosage was also investigated. Results show that the uptake of organic contaminant is directly proportional to the adsorbent loading, however this proportionality is only limited to below 150 g/l of Nyex after which the plot starts to gradually level off. The change in proportionality is due to a combination of the variation in concentration gradient and the coagulation of Nyex adsorbent at the basal plane.
4. Regeneration efficiency study showed that saturated Nyex adsorbent can be regenerated by electrochemical regeneration method with up to 100% regeneration efficiency.

5. Result also showed that a 4-log removal of biological contaminants in the form of E.coli and Pseudomonas can be achieved.

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