

A NOVEL ELECTROCHEMICAL ENHANCEMENT OF ACTIVATED CARBON FIBRE FOR THE PURIFICATION OF CONTAMINATED PHARMACEUTICAL EFFLUENTS

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ABSTRACT

Industrial discharge of pharmaceutical effluents has rapidly increased over the last decade and poses severe environmental threats. Activated carbon is the most prevalent adsorbent for wastewater treatment because of its efficiency and scalability for commercial usage. However, its application in treating pharmaceutical wastewater is unsustainable due to several pharmaceutical organics such as acetone that are harder to purify. To overcome this limitation, the electrochemical enhancement of ACF for the treatment of acetone-contaminated pharmaceutical wastewater was explored. It was found that at an applied voltage of -1.0V, electro-sorption at an optimum electrolyte concentration of 0.001M increased ACF's adsorption capacity for acetone by up to 107%. An improvement in adsorption capacity after electrochemical enhancement was consistent across a wide range of wastewater salinities. Freundlich and Langmuir isotherm studies also revealed that the maximum adsorption capacity of electrochemically enhanced ACF greatly outperformed that of unenhanced ACF and other well-known adsorbents. When electrochemically regenerated, ACF retained over 90% of its original adsorption capacity while the unenhanced ACF lost 39% of its initial adsorption capacity over three back-to-back cycles of adsorption and regeneration. Therefore, electrochemical enhancement increases the reusable lifespan of ACF, decreasing the amount of new ACF needed during treatment. Furthermore, electro-sorption and electrochemical regeneration have no lag time between each process, potentially allowing for a cyclic treatment process. This was explored in the construction of a working prototype. Electrochemically enhanced ACF holds immense promise as an effective and waste-minimizing technology for pharmaceutical wastewater purification.

1. INTRODUCTION

In recent years, increased demand for healthcare has accelerated the growth of the pharmaceutical industry. Singapore's vibrant pharmaceutical sector has seen rapid growth over the past half-century due to several factors, including a growing population and the development of new and innovative drug therapies to treat infectious and chronic conditions (Ho, 2020). Globally, each and every individual that requires medication - a universal necessity

- contributes to total water expenditure during pharmaceutical production, and its subsequent disposal as wastewater. Pharmaceutical manufacturing processes are batch and multi-stage, thus leading to generation of a huge quantity of effluent wastewater (Larsson et al., 2007). These pollutants pose a dire threat to the environment and even to human health.

Acetone is a prominent example of the organic pollutants found in pharmaceutical wastewater. Acetone, a polar organic with a low molecular weight, is often used as a solvent in pharmaceutical manufacturing (Gadipelly et al., 2014). For example, acetone is used as a solvent in the wet granulation process in the formulation of tablets or capsules (Grobowska & Parczewski, 2010). Acetone is highly volatile at room temperature and causes acute or chronic poisoning to humans by prolonged contact. In high concentrations, acetone is also toxic to wildlife and contributes to air pollution (Tang et al., 2016).

Conventional purification methods, such as reverse osmosis and electrochemical oxidation, are costly (Foo & Hameed, 2009) and unsustainable for the prolonged treatment of pharmaceutical wastewater. Activated carbon is widely hailed as the most prevalent adsorption material used for the removal of toxic organics from wastewater due to its effectiveness, capacity and scalability for commercial usage. In particular, activated carbon fibres (ACFs) which are a hybrid of carbon fibre and activated carbon have gained prominence. ACF is a promising microporous material with a fibre shape and well-defined porous structure. It has a high specific surface area ($800\text{-}3000\text{ m}^2/\text{g}$), fast adsorption and desorption rate and is easy to regenerate (Wang et al., 2018).

However, activated carbon has several limitations during the treatment of pharmaceutical wastewater. Activated carbon, being a non-polar adsorbent, is less efficient in its adsorption of polar organics with a low molecular weight such as acetone, many of which are present in pharmaceutical wastewater (Han et al., 2008). This causes activated carbon adsorbents to be quickly saturated with organic pollutants found in wastewater and lose their adsorption ability. Commercial chemical and thermal regeneration efforts of activated carbon for its reusability are also expensive and time consuming. Furthermore, activated carbon is largely produced from the carbonization of lignocellulosic materials (Menendez & Martin, 2006), such as rice husk and coal. Large-scale production of activated carbon depletes natural resources and is expensive. An estimated 300,000 tonnes of activated carbon is disposed yearly as secondary waste (Li et. al., 2008).

Electro-sorption (also known as electrochemical enhancement) has attracted attention as a promising technology that is primarily known to effectively desalinate seawater using activated carbon electrodes. Upon the passage of a potential difference through electrically conductive adsorbents, the adsorption capacity for ionic salts and heavy metal ions was enhanced by over 40% (Han et al., 2008). Recent forays have found that applying a second voltage on the activated carbon after adsorption could potentially enhance its regeneration for future reuse. By applying the use of electro-sorption for pharmaceutical wastewater purification, these enhancements could potentially address the limitations of activated carbon, allowing it to remain effective even during the purification of contaminated effluents with polar and low molecular weight pollutants.

Despite the urgent need to develop more efficient technologies for pharmaceutical wastewater purification, there are currently, to this study's knowledge, no studies on the electro-sorption of pharmaceutical pollutants. Electro-sorption has the unique features of low cost, is environmentally friendly, does not cause pollution and is simple to operate (Wang et al., 2012), making it a suitable technology for pharmaceutical wastewater treatment. Acetone as a pharmaceutical pollutant is uniquely both volatile and resistant to biodegradation, suggesting a need for greater understanding of new available methods in order to efficiently remove it from pharmaceutical effluents.

This study aims to explore and fill up this lack of material in the current field. By utilizing a novel electrochemical enhancement of activated carbon fibre (ACF), this study seeks to enhance ACF's performance as an attractive, versatile and effective treatment technology for the purification of pharmaceutical effluents.

OBJECTIVES

The scope of this study addresses the issue of pharmaceutical wastewater pollution, which is not only relevant to the local context of Singapore, but is a global problem affecting millions around the world. This study explores the use of electro-sorption and electrochemical regeneration to increase the adsorption capacity and reusability of ACF for acetone, a pharmaceutical pollutant which has relatively high polarity and low molecular weight. The maximum adsorption capacity and treatment mechanisms of ACF and electrochemically enhanced ACF would be determined through isotherm studies. The study also encompasses the integration of electro-sorption and electrochemical regeneration into a working prototype, which can be designed using automated technology to enable a cyclic treatment process.

HYPOTHESIS

It is hypothesized that electro-sorption is able to enhance the adsorption capacity of ACF for acetone across a range of wastewater salinity. The maximum adsorption capacity of electrochemically enhanced ACF will be higher than that of ACF. Electrochemical regeneration will also greatly improve the reusability of ACF.

2. MATERIALS AND METHODS

2.1 Materials

Acetone was procured from Merck while sodium sulfate was purchased from GCE Chemicals. ACFs were purchased from Guangdong, China.

2.2 Removal of acetone by ACF and electrochemically enhanced ACF

1.3 grams of ACF was suspended by a working electrode holder in 250ml solution containing acetone (1000 mg/L) and with varying concentrations of sodium sulfate (0.0001M, 0.001M, 0.01M).. A voltage of -1.0V was applied onto ACF using a potentiostat. An AgCl reference electrode was used to detect voltage applied, while a platinum plated electrode (PPE) served as the auxiliary electrode, completing the circuit (Figure 2). Electro-sorption was conducted for 40 minutes, where adsorption reached equilibrium.

A similar setup without voltage applied across ACF served as the control. To account for volatilization of acetone, a setup with neither ACF nor voltage applied was employed. UV-Vis spectroscopy at a wavelength of 267nm was used to determine the final concentration of acetone after adsorption and electro-sorption. Cyclic voltammetry experiments were done to verify that acetone is not reduced or oxidised during electro-sorption (Appendix B, Page 15).

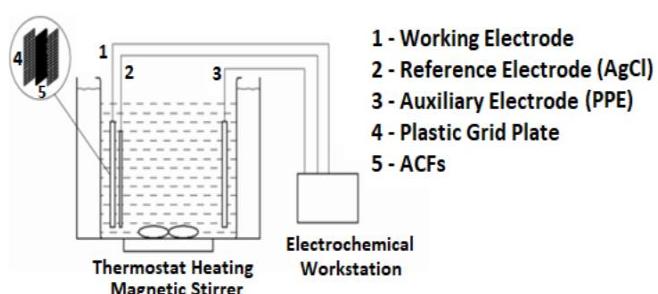


Figure 1: Schematic of electro-sorption setup



Figure 2: Arrangement of electrodes

2.3 Isotherm Studies on ACF and electrochemically enhanced ACF

1.3g of ACF and electrochemically enhanced ACF were submerged in a setup similar to the one mentioned in section 2.2, with a salinity of 0.001M sodium sulfate and varying acetone concentration (400ppm, 600ppm, 800ppm, 1000ppm and 1200ppm).

The adsorption capacity at equilibrium, Q_e , in mg/g was calculated with the formula:

$$Q_e = \frac{(C_i - C_e) \times V}{m}$$

C_i is the initial acetone concentration in mg/L, C_e is the concentration of acetone at equilibrium in mg/L, V is the volume of the solution in litres and m is the mass of the ACF in grams. These were then used to calculate Q_{max} , the maximum adsorption capacity. R^2 , which is the coefficient of determination, was compared between the Langmuir and Freundlich isotherms to greater understand the mechanisms of adsorption and electro-sorption by ACF (Appendix A, Page 13 -14).

2.4 Reusability Studies on ACF

After adsorption or electro-sorption, ACFs from both set ups were submerged in 100ml of 0.001M sodium sulfate solution. A voltage of +0.6V was applied on the electrochemically enhanced ACF for 30 minutes. ACF and electrochemically enhanced ACF was subjected to three consecutive cycles of adsorption/electro-sorption and regeneration/electrochemical regeneration with their adsorption capacity at the end of each cycle determined.

3. RESULTS AND DISCUSSIONS

3.1 Removal of acetone by ACF and electrochemically enhanced ACF

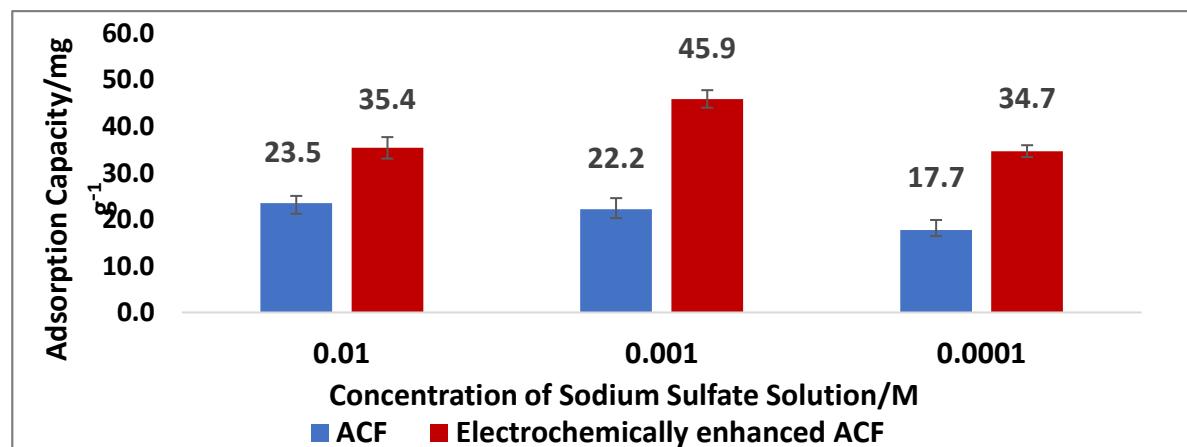


Figure 3: Removal of acetone by ACF and electrochemically enhanced ACF. Data represents mean adsorption capacity \pm SE of triplicates.

A wide range of salt concentration is naturally found in pharmaceutical wastewater. Figure 3 shows that the electrochemically enhanced ACF removed more acetone than the unenhanced ACF across all wastewater salinities, with the greatest increase being 107% when sodium sulfate concentration is 0.001M. Similar to ACF, the adsorption capacity of electrochemically enhanced ACF increased as salinity increased from 0.0001M to 0.001M of sodium sulfate. This is likely due to the “salting out” of acetone (Matkowich & Christian, 1973) where the solubility of acetone decreases as more sodium sulfate is dissolved in the solution, resulting in more acetone being retained on the surface of ACF. However, at 0.01M of sodium sulfate, the adsorption capacity of electrochemically enhanced ACF decreases, as the excess ions occupy adsorption sites on ACF due to electrostatic attraction from the voltage applied (Wang et al., 2014).

It is proposed that ACF adsorbs acetone due to permanent dipole-permanent dipole interactions and hydrogen bonding between its surface functional groups and acetone molecules (Figure 4).

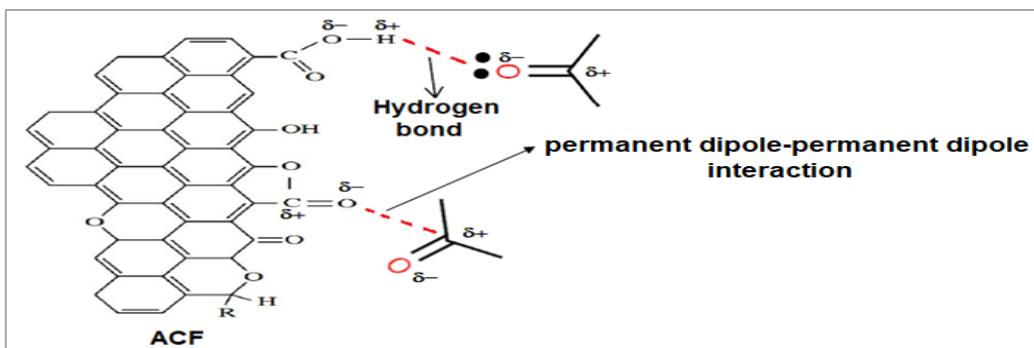


Figure 4: Proposed adsorption mechanism of acetone by ACF

To explain the increase in adsorption capacity, Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS) analysis were conducted on ACF before and after electro-sorption. Both SEM images in Figures 5 and 6 were performed at an acceleration voltage of 5.0 kV and aperture working distance of 8.0 mm.

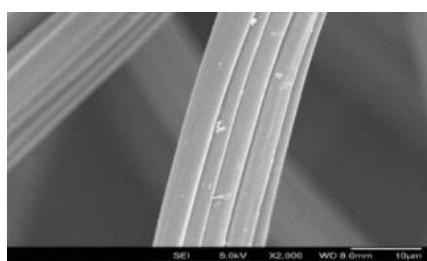


Figure 5: SEM of ACF before electro-sorption at 2000x magnification

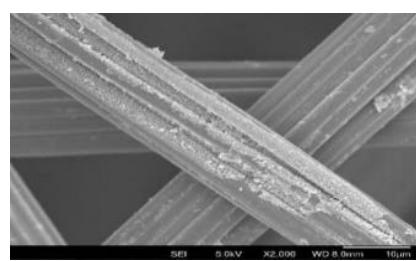


Figure 6: SEM of ACF after electro-sorption at 2000x magnification

Figures 5 and 6 reveal that ACF after electro-sorption has a rougher and more uneven surface, possibly due to electrochemical polarization, which is attributed to electrostatic repulsion between surface functional groups (Su & Hatton, 2007), inducing inelastic deformation on the surface of the ACF. This increases the number of adsorption sites available on ACF's surface, enhancing its adsorption capacity.

EDS data also suggests an increase in the oxygen containing functional groups of ACF after electro-sorption (Table 1), which improves the hydrophilicity of ACF, thereby allowing ACF to better adsorb acetone via hydrophilic interactions. At a negative voltage applied during electro-sorption, active oxidising free hydroxyl radicals are generated on the surface of the ACF, causing it to be oxidized (Han et al., 2006; Han et al., 2007).

Table 1: EDS of oxygen content/% in unenhanced ACF and ACF after electro-sorption

Oxygen content in unenhanced ACF/%	Oxygen content in ACF after electro-sorption
6.71	9.70

3.2 Isotherm Studies

The equilibrium concentration data of ACF and electro-sorption were fitted into the Langmuir and Freundlich isotherms (Appendix A, Page 13-14). Table 2 reveals that electrochemically enhanced ACF not only has a much higher maximum adsorption capacity (Q_{\max}) than unenhanced ACF, it also outperforms other adsorbents like zeolite and silica.

Table 2: Q_{\max} derived from Langmuir isotherm plots of electrochemically enhanced ACF, unenhanced ACF and other adsorbents for acetone adsorption

Adsorbent	Q_{\max} /mg g-1	Sources
Enhanced ACF	189	This study
Unenhanced ACF	50	This study
Zeolite	57	(Zeng & Bai, 2016)
Silica	68	(Lin, Bai & Chang, 2012)

By comparing R^2 (Coefficient of determination) of isotherm plots, mechanisms of adsorption and electro-sorption of acetone were compared (Table 3). The Freundlich isotherm is a better fit for electrochemically enhanced ACF as its R^2 is greater, suggesting multilayer

and heterogeneous adsorption. In contrast, adsorption by unenhanced ACF better fits the Langmuir isotherm, suggesting that adsorption is predominantly monolayer and homogeneous.

Table 3: R^2 of Freundlich and Langmuir plots of electrochemically enhanced ACF and unenhanced ACF for the adsorption of acetone

Adsorbent	R^2 (Freundlich)	R^2 (Langmuir)
Enhanced ACF	0.994	0.952
ACF	0.864	0.880

3.3 Regeneration and Electrochemical Regeneration of ACF

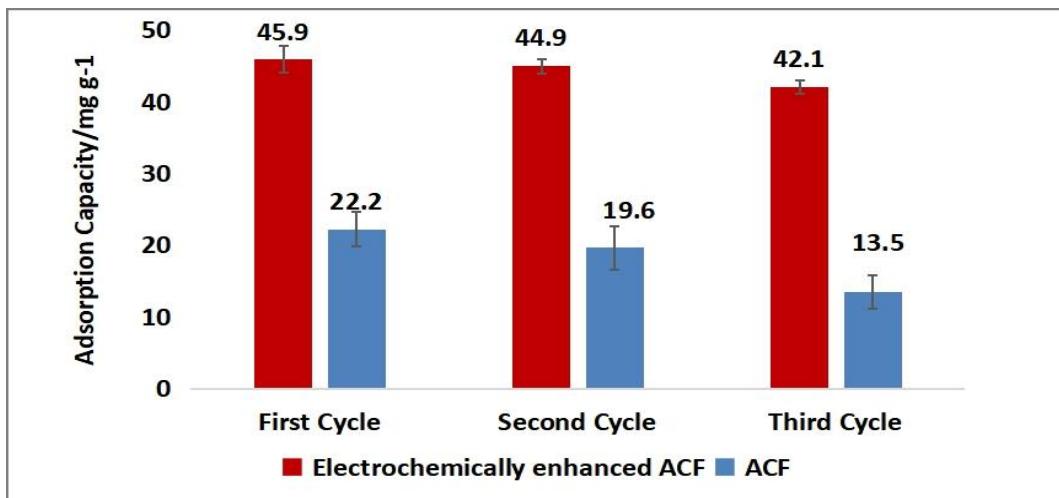


Figure 7: Regeneration and Electrochemical Regeneration of ACF

Figure 7 reveals that after three cycles of adsorption and regeneration, the electrochemically enhanced and regenerated ACF maintained over 90% of its original adsorption capacity, while ACF lost about 39% of its initial adsorption capacity by the third cycle. Therefore, electrochemical enhancement increases the reusable lifespan of ACF, decreasing the amount of new ACF needed during wastewater treatment. Under a positive voltage applied during regeneration, H^+ ions are produced at the ACF functioning as an anode, causing local pH to decrease (Mehta & Flora, 1997). As H^+ ions protonate acetone, the hydrogen bonds between acetone and ACF are weakened, promoting the desorption of acetone into the solution, rendering the regeneration of ACF efficient.

Notably, electro-sorption was carried out immediately after regeneration, with no lag time in between cycles. This suggests that electro-sorption and electrochemical regeneration can be conducted immediately after each other, rendering a cyclic process. In comparison, commercial chemical regeneration of activated carbon typically requires up to several hours

between cycles for the drying of adsorbents (Han et al., 2006). Thermal regeneration, a widely used commercial regeneration method, is energy intensive as it requires high temperatures up to 800°C during its pyrolytic stage (Foo & Hameed, 2009). In contrast, electrochemical regeneration, which has a low energy consumption as it operates in low direct currents (Foo & Hameed, 2009), is more cost-effective than conventional chemical or thermal regeneration.

3.4 Construction of Prototype for Integrated Treatment of Pharmaceutical Wastewater

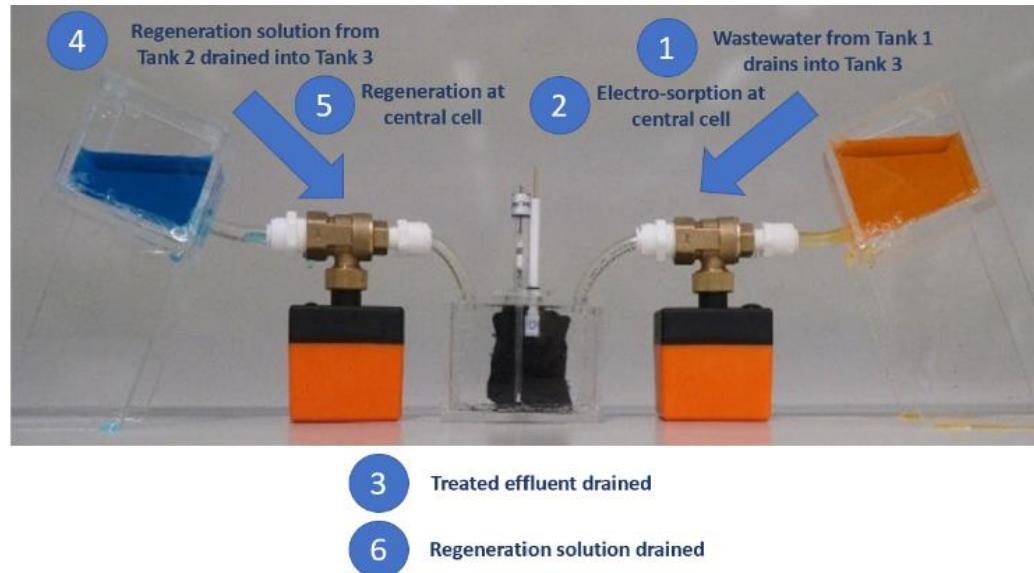


Figure 8: Prototype constructed

A prototype was designed for the large-scale treatment of pharmaceutical wastewater. The prototype consists of three containers, two of which are containers for the wastewater and regeneration solutions with the third being the central electrochemical cell. The containers were constructed using 5.0 mm thick acrylic sheets cut out using a laser cutter joined together with acrylic glue. A 10 mm hole was drilled in each of the reagent containers to attach a pipe by which the container would be drained. In the electrochemical cell, a 40 mm circle was cut out from the top to fit the electrodes in. To control the flow of solution, two electronic ball valves were installed at the reagent containers. A third ball valve was installed at the bottom of the electrochemical cell to drain away the solution after electro-sorption and regeneration cycles. With the use of the electronic ball valves, the process is fully automated.

With the flick of a button, wastewater from Tank 1 drains into the central cell (Tank 3) where electro-sorption of pharmaceutical wastes occurs. After electro-sorption, the treated effluent will be drained. Regeneration solution from Tank 2 will then be channelled into the central cell to desorb the adsorbed pollutants so that the ACF could be regenerated. After

desorption, the spent regeneration solution will be drained, and the cycle repeats. The process is automated, rapid and efficient. This can be visualized in Figure 8.

The prototype demonstrates the potential for a cyclic treatment process, which could allow for a single batch of ACF to potentially treat tonnes of pharmaceutical wastewater without human intervention. It also minimizes lag time between electro-sorption and electrochemical regeneration, maximizing efficiency. In an industrial setting, the prototype can be upscaled, rendering treatment of pharmaceutical wastewater by electro-sorption and electrochemical regeneration even more attractive and convenient.

4. Conclusions and Future Work

Electro-sorption successfully increases the adsorption capacity of ACF on acetone up to 107% across a wide range of wastewater salinity, while increasing the maximum adsorption capacity of ACF from 50 mg g^{-1} to 189 mg g^{-1} . Furthermore, electrochemical regeneration can retain over 90% of ACF's adsorption capacity over three continuous cycles of adsorption and regeneration. The cyclic nature of electro-sorption and electrochemical regeneration allows for a rapid, simple and convenient treatment process. Electro-sorption and electrochemical regeneration hold immense promise to propel adsorption as a more cost-efficient and waste minimizing option for pharmaceutical wastewater purification.

A model on how electrochemically enhanced ACF can be used in industrial wastewater treatment plants has been proposed and constructed. The integration of electrochemical regeneration and electro-sorption *in situ* could effectively reduce labour costs. On an industrial scale, such a prototype could potentially be used for a large-scale cyclic treatment process for acetone-contaminated pharmaceutical wastewater, allowing for a facile treatment process.

In the future, the study could be extended to include a wide range of ions and other pharmaceutical solvents such as ethylene glycol and dimethyl sulfoxide. A synthetic form of pharmaceutical wastewater containing a mixture of these solvents, together with a high concentration of ions such as Cl^- and NO_3^- could be used to mimic real-life pharmaceutical wastewater (Gadipelly *et al.*, 2014), so as to ensure that electrochemically enhanced ACF is effective and widely-applicable even during real life wastewater treatment. Studies could also be conducted to determine the maximum amount of times electrochemically enhanced ACF can be reused while retaining over 90% of its adsorption capacity, as the current study only investigated this up to three times.

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Appendix A: Adsorption Isotherms of Acetone by ACF and electrochemically enhanced ACF

The final concentration data (C_e) from isotherm studies on acetone with ACF and electrochemically enhanced ACF was used to calculate adsorption capacity, Q_e . Both were fitted into the Freundlich and Langmuir isotherms. The maximum adsorption capacity (Q_{max}),

favourability of adsorption ($\frac{1}{n}$), and K_f , which is related to maximum sorption capacity were then derived.

The Langmuir isotherm, which assumes monolayer adsorption onto a homogenous surface,

was plotted using the linearized equation: $\frac{C_e}{Q_e} = \frac{1}{bQ_{max}} + \frac{C_e}{Q_{max}}$, where C_e is the final concentration of acetone at equilibrium in mg/L, Q_e is the adsorption capacity at equilibrium in mg/L, Q_{max} is the maximum adsorption capacity and b is a constant. The inverse of the gradient can be used to derive Q_{max} .

The Freundlich isotherm, in contrast to the Langmuir isotherm, assumes multilayer adsorption onto a heterogeneous surface. It was plotted using the linearized equation:

$\lg Q_e = \lg K_f + \frac{1}{n} \lg C_e$, where C_e is the final concentration of the acetone at equilibrium in mg/L, Q_e is the adsorption capacity in mg/g at equilibrium, K_f is a constant proportional to the maximum adsorption capacity and $\frac{1}{n}$ is another constant which determines favourability of adsorption.

The respective isotherms determined are shown below:

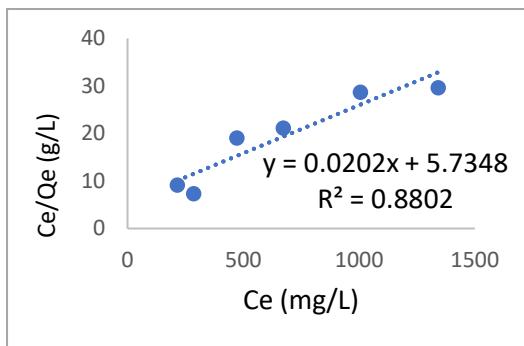


Figure 9: Langmuir isotherm for ACF

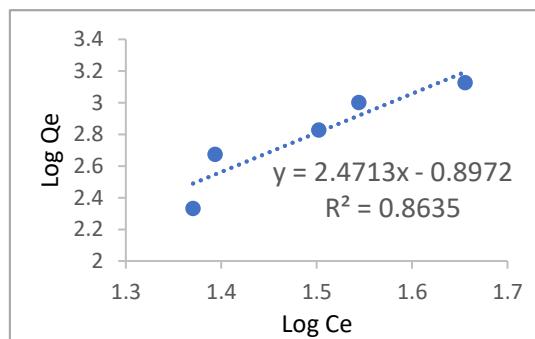


Figure 10: Freundlich isotherm for ACF

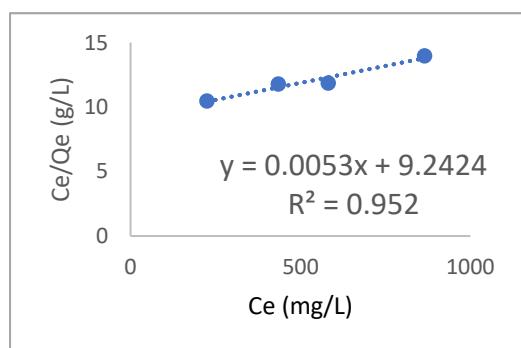


Figure 11: Langmuir isotherm for electrochemically enhanced ACF

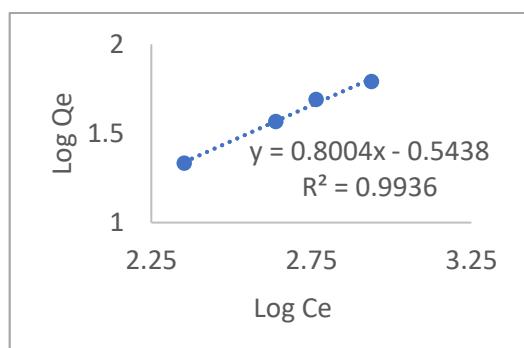


Figure 12: Freundlich isotherm for electrochemically enhanced ACF

Appendix B: Cyclic Voltammetry

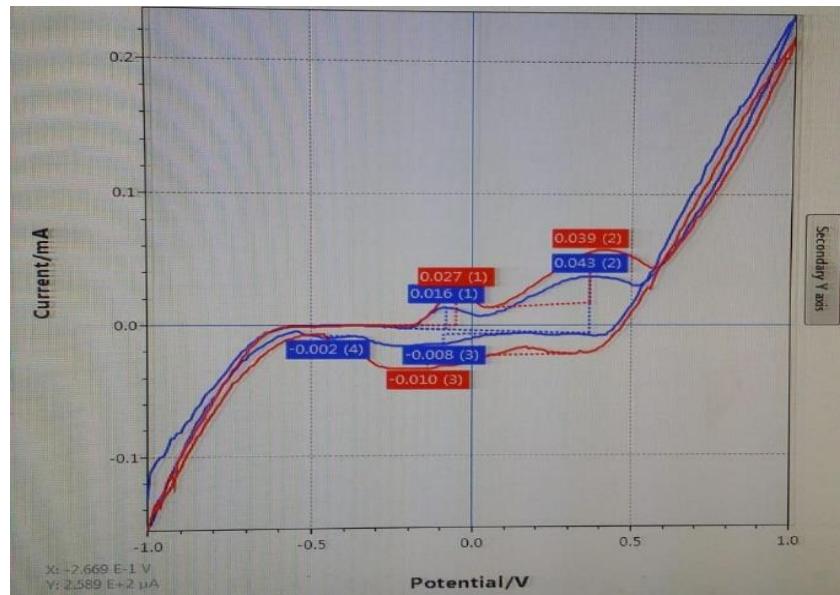


Figure 13: Cyclic voltammetries of acetone solution and saline solution

In order to ensure that acetone was not oxidized or reduced during the electrosorption process, the voltage applied on ACF during electrosorption must be controlled in the range of redox potentials. Figure 13 shows the cyclic voltammetries of the solution with and without acetone at scan rate of 50 mV/s, which were measured with a potentiostat. The red line represents the cyclic voltammetry of a 0.001M sodium sulfate solution while the blue represents the cyclic voltammetry of a 1000 mg/L acetone solution with 0.001M sodium sulfate. Comparing the cyclic voltammetry of the solution with and without acetone, it was found that there are no additional peaks between -1.0V and +1.0V, indicating that acetone is not reduced or oxidized in this range of voltage applied.