Adsorption of Perfluoroalkyl Compounds onto Microporous Agave Sisalana Activated Carbon Fibre

Serge M. Imwer, John B.N. Mudumbi, Seteno K.O. Ntwampe, Elizabeth I. Omodanisi, Adegbenro P. Daso, Marshall S. Sheldon and Sebusi Odisitse

Abstract— Perfluoroalkyl compounds (PFCs) have been shown to cause harmful effects on the environment and human health. A major concern is their wide range of uses in consumer products which have been detected in food and drinking water. The study reported the removal of these contaminants using a microporous activated carbon fibre (ACF) made from an indigenous plant, *Agave sisalana*, widely available across sub-Saharan Africa, by using electro-physicochemical methods. An electro-physicochemical adsorption regime was designed, to facilitate the rapid adsorption of PFOS and PFOA from contaminated drinking water in an electrolytic cell. Initially, adsorption studies (n = 48) using sonication (20 kHz) in batch systems indicated efficient removal of PFOA and PFOS within 120 min, with numerous samples (n = 14) achieving complete removal for both PFOA and PFOS. The minimum removal rates observed were 65.55% for PFOA and 95.92% for PFOS.

Keywords— activated cacbon fibre, Adsoption, *Agave sisalana*, Perfluoroalkyl compounds.

I. INTRODUCTION

Perfluorooctanoic acid (PFOA) and perfluorooctane sulfonate (PFOS) are inert chemical compounds that have been determined to be bio-accumulative, persistent and major environmental pollutants in water. They have been used in many industrial processes and are found in Teflon-based cookware, drinking tap water and various media (Herrera & Alvarez, 2008). As these compounds are highly soluble in water, they contaminate environmental water sources, that is, ground and surface waters (Yu et al., 2009). To date, thousands of PFCs have been discovered, but PFOA and PFOS have been reported to be the most harmful to humans (OECD, 2002). PFCs have extensively been used as lubricants, surfactants, fire retardants, adhesives, paper-coats, refrigerants, propellants and agro-chemicals even with their detrimental effect on humans and the environment.

The unique properties of PFOA and PFOS, which make them suitable for various industrial applications and non-biodegradability, are the lack of carbon-hydrogen (C–H) bonds in their structure, which are replaced by carbon-fluorine (C–F) bonds (Renner, 2001; Qiu, 2007). As a result, these compounds are thermodynamically stable, which makes them non-biodegradable.

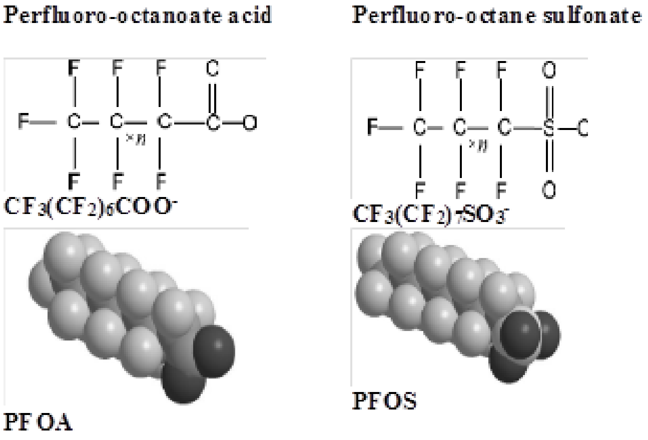

Fig. 1. Structure of PFOA and PFOS (Qiu, 2007)

Their presence in the environment has been reported in several countries (Rovira et al., 2019; Torre et al., 2019) and recently in South Africa (Mudumbi, 2013). For example, in a study conducted in the USA, the presence of PFOA in potable water in Washington and Virginia was determined to be in the range of 1.9 to 4.9 ng /L (Bartell *et al.*, 2010), with the median

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S.M Imwer was with the Bioresource Engineering Research Group (*BioERG*), Department of Biotechnology, Faculty of Applied Sciences, Cape Peninsula University of Technology, Keizersgracht and Tennant Street, Zonnebloem, P.O. Box 652, Cape Town, 8000, South Africa.

J.B.N. Mudumbi is with the Bioresource Engineering Research Group (BioERG), Faculty of Applied Sciences, Department of Biotechnology, Cape Peninsula University of Technology, Keizersgracht and Tennant Street, Zonnebloem, P.O. Box 652, Cape Town, 8000, South Africa.

S.K.O. Ntwampe is the founder of Bioresource Engineering Research Group (BioERG), Faculty of Engineering and the Built Environment, Department of Chemical Engineering, Cape Peninsula University of Technology, PO Box 1906, Bellville, 7535, South Africa.

E.I. Omodanisi is with the Bioresource Engineering Research Group (BioERG), Department of Biotechnology, Faculty of Applied Sciences, Cape Peninsula University of Technology, Keizersgracht and Tennant Street, Zonnebloem, P.O. Box 652, Cape Town, 8000, South Africa.

A.P. Daso is with the Department of Environmental, Water and Earth Sciences, Faculty of Science, Tshwane University of Technology, Pretoria 0083, South Africa.

M.S. Sheldona is with the Faculty of Engineering and the Built Environment, Department of Chemical Engineering, Cape Peninsula University of Technology, PO Box 1906, Bellville, 7535, South Africa.

S. Odisitse is with the Faculty of Science, Department of Chemistry and Forensic Sciences, Botswana International University of Sciences and Technology, P/bag x16, Palapye, Botswana, BW

PFOA concentration in human sera being 8.75 ng/L (range 4 to 21 ng /L) (Steenland *et al.*, 2010), indicating a possible continuous exposure or ingestion. Similarly, the presence of PFOS and PFOA was detected and quantified in tap water in China, with the concentration range of <0.1 to 14.8 ng/L and <0.1 to 45 ng/L for PFOS and PFOA, respectively (Jin *et al.*, 2009).

A similar survey was done in South Africa (SA), where the presence of PFOS and PFOA in maternal serum and cord blood of South African women was determined in a range of 0.5 to 16 ng/mL and 0.4 to 8.5 ng/mL, respectively (Hanssen *et al.*, 2010). This prompted a study to assess PFOA and PFOS concentrations in the drinking water of the Western Cape, South Africa (Booi, 2013). The results indicated a widespread contaminate of PFOA and PFOS in drinking water, including river water used to irrigate agricultural produce (Mudumbi, 2013). The major route of these pollutants in terms of environmental contamination is through contaminated water sources such as wastewater discharged by industries which have to apply products containing these substances during the manufacturing process. Several studies have been done to assess their prevalence in natural water sources such as river-, sea-, and rainwater (Melzer *et al*., 2010). Similarly, Skutlarek et al. (2006) carried out a study in two German rivers; Rhine River and Moehne River, canals and drinking waters of the Ruhr catchment area to quantify the concentration of perfluorinated surfactants in surface and drinking waters, where 12 different PFCs, including PFOA and PFOS, were found.

Various studies have published information on the removal of PFOS and PFOA by adsorption. For instance, a comparison was made whereby powdered activated carbon (PAC), granulated activated carbon (Ounas *et al.*, 2009), and ion exchange resin (AI400) were evaluated for the removal of PFOA and PFOS from water. It was found that the adsorbent size influenced the sorption kinetics with activated carbon (AC) being effective in comparison with ion exchange (Yu *et al.*, 2009).

Agave sisalana (sisal) is a tropical plant which falls under the genus *Agave*. This plant is principally monocarpic (i.e. reproducing only once after several years and then dying). Thus, *A. sisalana* is of considerable ecological (as keystone species) and economic importance as it is used as a raw material for several industries in various countries (Good-Avila *et al.*, 2006). It is an important cordage fibre-yielding plant that is drought resistant and can withstand extreme dry conditions. Studies have shown that sisal fibres can be carbonised to produce a silver supported activated carbon fibre (Chen *et al.*, 2005). Similarly, sisal waste was also used as a feedstock to produce ACF by a chemical activation process with K_2CO_3 as the activating agent under N_2 flow (Mestre *et al.*, 2011). The result revealed that adsorption of PFOA and PFOS on ACF was a monolayer adsorption type phenomenon.

The study evaluated the the removal of PFOS and PFOA from contaminated water using activated carbon fibre obtained from *Agave sisalana*.

II. MATERIALS AND METHODS

A. Samples Collection and Processing

This process was performed according to previous studies (Conter, 1903; Chen et al., 2005). As such, after sisal reached maturity (3 years), the lower and older leaves were removed close to the head, and scraped one by one, using a scraper. The fibres were extracted, cleaned and then dried. The drying process consisted of hanging the fibres in the sun, to allow them to lose their lustre. Nevertheless, the fibres were kept straight and cleaned to avoid mixing of the fibres and tuft.

B. Manufacturing of activated carbon fibre (ACF)

Different reagents were sprayed onto the sisal fibres. After spraying along the fibre length, samples were placed in an oven at a temperature of 110°C for about an hour until completely dry. The process was repeated until a total volume of 10 ml of the reagent was sprayed. Long sisal fibres were then cut to a length of 3 mm, loaded into ceramic boats, packed in a tubular furnace and pyrolysed at a required temperature under nitrogen (N_2). However, since the carbonization and the chemical activation were combined, the purging gas was switched to carbon dioxide (CO_2) when the activation temperature was reached.

Once activation had taken place, a period of time elapsed, the temperature was then reduced under N_2) to room temperature. The ACF obtained was then washed with 6 L of warm distilled water. During the washing process, bromothymol blue was used as an indicator to assess the neutrality of the recovered rinsing water. Samples were then dried in an oven overnight. The final product (ACF) were sealed in a bag (5 x 6 cm) using heat at a varying temperature from 450-650 °C. The concentration of the activation reagents is shown in Table 1, while Figure 2.

TABLE 1: CONCENTRATION OF DIFFERENT REAGENTS USED FOR THE ACTIVATION PROCESS

Activation agent	Concentration				
KOH	21.6 M	4.32 M	2.16 M	1.08 M	0.54 M
NaOH	25 M	5 M	2.5 M	1.25 M	0.625 M
$ZnCl_2$	31.8 M	6.36 M	3.18 M	1.59 M	0.795 M
H_3PO_4	14.6 M	2.92 M	1.46 M	0.73 M	0.365 M

Control pure sisal/pyrolysed sisal: KOH-121 g/100 ml at 25°C (saturated); NaOH - 100 g/100 ml at 25°C (saturated); $ZnCl_2$ 432 g/ 100 ml at 25°C (saturated); H_3PO_4 - Used as is and the dilution of 1/5, 1/10, 1/20, 1/40 thereafter for all of them.

A. Preparation of PFOS and PFOA contaminated water

A 2 L polypropylene (PP) container was washed with methanol, to avoid any PFC contamination, then rinsed with Milli-Q water (Mastalerz *et al.*, 2011). Two litres of distilled water were placed in 2 L PP bottle; to which 0.1 μL of PFOA and PFOS, at the concentration of 2 g/L each, was added into the distilled water to produce a solution of polluted water at a concentration of 100 ng/L of PFOA and PFOS. The synthetic water used, was based on raw and treated drinking assessments from another study (Booi, 2013), whereby PFC concentrations were determined in the range up to 40 ng/L and 100 ng/L observed by Mudumbi *et al*. (2014), in river water, for both PFOA and PFOS.

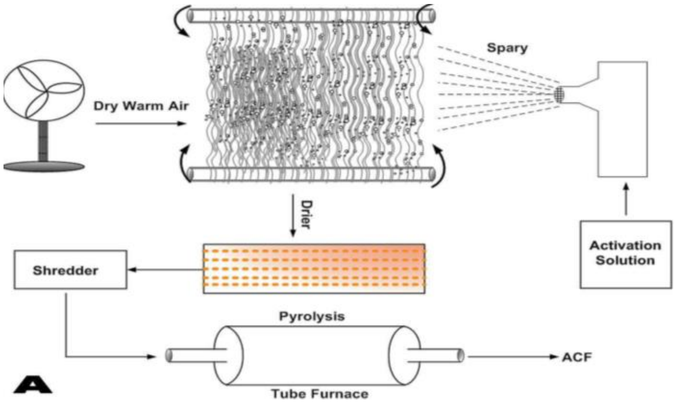

Fig. 2. Schematic representation of the preparation method for ACF using *Agave Sisalana*

B. *Surface area characterization*

Analyses were done on a 3Flex Surface Characterization Analyzer from Micromeritics Instrument Corporation. Prior to analysis, the samples were degassed using a Vacprep 061 at 250°C for 24 hours under vacuum (Webb & Orr, 1997). CO_2 was used as the adsorbate gas at a temperature of 0°C. The different models used for surface characterization and the surface area obtained using different feedstock can found in previous work (Imwer, 2014). The surface area models used tend to over-estimate surface properties during analysis – (Results), therefore, the specific surface area for the ACF was quantified using the Dubinin-Astakhov isotherm with a relative pressure (P/P_0) up to 0.01. The sample mass was between 0.2 to 0.3 g.

C. *Adsorption studies*

1) *Physico-chemical assisted adsorption studies*

Physico-chemical assisted adsorption was performed using 150 ml of contaminated water in a 250 mL polypropylene (PP) beaker without pH adjustment; then an ACF bag containing 0.57 g of *A. Sisalana* was immersed into the polluted water with subsequent sonication. The sonicator was set at 20 kHz, whichprobe was rinsed with 99% methanol and Milli-Q water after each experiment before processing new samples. The samples were sonicated for 2 hours. Thereafter, the ACF bags were dried in the oven at 70°C, while a 10 ml solution was taken from the bulk solution for analysis. The sample solution for analysis was filtered (0.22 μm) and stored in the refrigerator at 4 °C prior the analysis, which was performed using a liquid chromatography system, combined with tripartite quadrupole linear ion trap tandem mass spectrometer (LC-MS/MS), but only after Solid Phase Extraction (SPE) was executed. LC-MS/MS was performed on a Ultimate 3000 Dionex HPLC system (Dionex Softron, Germering, Germany), equipped with a binary solvent manager and auto sampler, coupled to a Brucker ESI Ion Trap Mass Spectrometer (Bruker Daltonik GmbH, Germany). The products were separated by reversed-phase chromatography on a Waters Sunfire C18 column 5 μm, 4.6 × 150 mm (Dublin, Ireland) using gradient elution at a flow rate of 0.8 ml min^{-1}, an injection volume of 10 μL and an oven temperature of 30°C. The gradient was set up as follows: 64 % A to 44 % A (12 min); 44 % A to 1 % A (12– 13 min); 64 % A (13.1–20 min); solvent A – 5mM ammonium acetate in water, solvent B – acetonitrile. MS spectra were acquired in a negative mode using the full scan mode with dual spray for reference mass solution. Electrospray voltage was set to -3500 V. Dry gas flow was set at 9 l min^{-1} with a temperature of 350 °C and the nebuliser gas pressure was set to 347.96 KPa. The compounds were quantified using Quant Analysis software. The cumulative loading for the adsorption was determined using the mathematical expression in Eq. 1:

$$q_{st} = \frac{X}{m} = \frac{(C_i - C_s) * V_r}{volume\ conversion\ factor} \tag{1}$$

Where X – is the amount of PFOA/PFOS adsorbed onto the ACF, m – is the mass of the adsorbent (ACF), C_i – is the initial concentration of PFOA/PFOS in solution, C_s – is the amount of residual PFOA/PFOS in solution, V_r – is the volume of the PFOA/PFOS contaminated water used during adsorption, and q_{st} – is the cumulative loading parameter for PFOA/PFOS adsorption. This mathematical expression was also used for the determination of PFOA/PFOS adsorption using an Electro-physico-chemical method.

2) *Electro-physico-chemical assisted adsorption*

A neckless 250 mL PP bottle containing 150 mL of contaminated water was used as an electrolytic - type cell, where the electrodes were 55 mm distant from each other and the ACF bag containing 0.57 g of *A. Sisalana* being placed at the anode. The cathode was made of stainless steel while the anode was plated with a platinum layer. The direct current used for electrolysis was 12 volt (V). During electrolysis, sonication was also applied, with samples being taken every 20 minutes for a period of 2 hours.

III. RESULTS AND DISCUSSION

A. *Manufacturing of activated carbon fibre (ACF)*

Activated carbon fibre (ACF) was synthesised from Sisal fibres via the spraying technique. Fig. 3 represent a sample obtained using the spray method. Although the spray scattered the activation chemical solutions on the fibre, the erosive effect of the method suggested that intermittent application of the spray was necessary, with a drying effect for immediate drying of the droplets when they are on the surface of the sisal, resulting in the formation of pores. A comparative analysis was made using Scanning Electron Microscopy (SEM) monographs of the fibre obtained in this study with other SEM monographs reported in published works (Phan *et al.*, 2006; Tan *et al.*, 2007; Tan *et al.*, 2008). These samples were very similar, with the fibre obtained from this study showing a highly porous external and internal structure of the fibre.

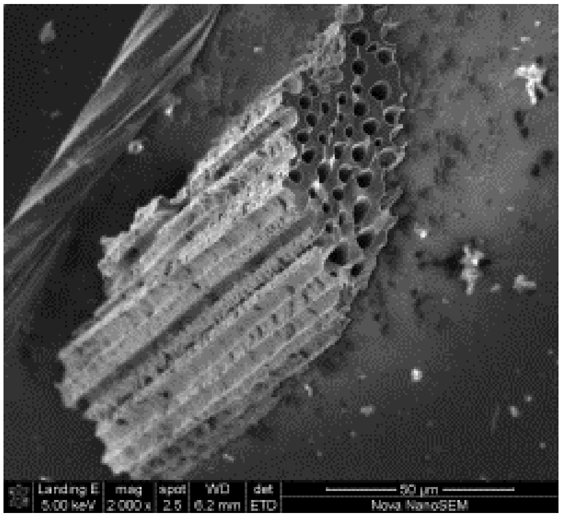

Fig. 3. SEM micrograph of sisal fibre after being pyrolysed by applying the activation reagents.

Table 2 how the activation reagent were selected, while listed the operating temperature of the furnace during ACF production and the yield obtained.

TABLE II: SELECTION OF THE OPTIMUM ACTIVATION AGENT

Parameters/Reagents		$^yZnCl_2$	$^zH_3PO_4$	^xKOH
% Removal	PFOA	94.2 – 100	65.55 – 84.75	75,57 – 99.19
	PFOS	98.4 – 99.7	95.92 – 98.79	98.74 – 99.74
Conditions ACF Production	Temperature	650 – 850	650 – 850	650
	Activation time	45 min – 3hrs 28 min	45 min – 3hrs	1hr 53 min
	Activation reagent conc. (M)	1.59	0.73	0.54
% Yield		32	32.7	33.8

y – Average of 6 samples, z - Average of 3 samples, w - Average of 1 sample and x -Average of 4 samples

B. *PFOA and PFOS adsorption onto AFC using sonication*

During the experiments, concentrations of PFOA and PFOS in the polluted water samples were 100 ng/L and 100 ng/L, respectively. Table 2 displays the experimental results obtained from the adsorption of PFOA and PFOS assisted with an ultrasound probe. These experiments were performed under the following conditions: duration: 120 min; amplitude: 20 kHz; power: 28W; 220 J, with the final temperature of the water being 35°C.

The ACF prepared under different pyrolysis conditions performed differently. However, the removal efficiency of PFOS was the most consistent, albeit available evidence reporting that PFOA is largely easier to be adsorbed than PFOS. However, in the present study, we strongly believe that sonication assists or plays a role in the physical attachment of a contaminant to a solid matrix. As such, since PFOA and PFOS could attach to the surface of the ACF by physical means, the same physical forces could also result in the adsorption-desorption cyclic phenomena, for which a contaminant with weak Van der Waal's forces could largely remain within the solution in which it was initially present. As previously explained, the formation of cavitations can, therefore, result in the desorption of the adsorped PFCs.

For this study, this removal of the two pollutants was not complete for both PFCs in the samples. This led us to presume that each ACF sample had a different affinity to PFOA and PFOS, perhaps with the reagent used for its activation playing a major role.

C. *Surface area determination*

With regard to the characterisation of ACF samples produced during this study, four models were used to evaluate the surface area, pore size and pore volume. These models were: (i) Brunauer-Emmett-Teller, (ii) Horvath–Kawazoe, (iii) Dubinin–Radushkevich and (iv) Dubinin–Astakhov. Based on the report of the analysis, it was found that the samples produced in this study had mostly micropores and almost no mesopores. Of the models mentioned above, the Dubinin–Astakhov isotherm was used to evaluate the micropore surface area for this study, compared with other studies where specifications were given of the stratification of AC pores, such as macropores (> 500 Å), mesopores (20 – 500 Å) and micropores (< 20 Å) (Pelekani & Snoeyink, 1999), with sizing related to the pore's diameter. In order to perform this analysis, low-pressure CO_2 adsorption at 0°C was used to describe the pore size and surface characteristics of the ACF. The Dubinin–Astakhov equation can be used to linearise adsorption data that generate curved Dubinin–Astakhov plots. Micropore volumes found using the Dubinin–Astakhov equation agreed with the total volume generated by the N_2 adsorption method which occurs at - 196°C for all activated carbon studies (Ghosal & Smith, 1996).

The ACF prepared with $ZnCl_2$ at the concentration of 1.59M had a micropore surface area of 1036 m^2/g with a limiting volume of 0.32 cm^3/g, while the one prepared with KOH at the concentration of 0.54M had a micropore surface area of $1285m^2/g$ with a limiting volume of 0.39 cm^3/g see Table 3. In a study on the production of activated carbon from olive industry waste using NaOH, Na_2CO_3, HCl, H_2SO_4, H_3PO_4 and KOH, the later (i.e. KOH) was reported to be the best activation reagent when it comes to the adsorption of trace contaminants in water (Ounas *et al.*, 2009). In this study, the focus was on ACF made from KOH, which was then used for, electro-physico-chemical adsorption kinetics.

TABLE III. PHYSICAL PROPERTIES OF THE *AGAVE SISALANA* ACF

Property	$ZnCl_2$ [1.59 M]	KOH [0.54 M]
Surface area (m^2g^{-1})*	1036.76	1285.83
Micropore volume (cm^3g^{-1})	0.097	0.13
Total pore volume (cm^3g^{-1})	0.32	0.39
Microporosity (%)	30.36	34.16
Average micropore width (Å)	(3.7 – 8.04)	(3.7 – 8.21)

*Determined using Dubinin – Astakhov Isotherm

Activated carbon fibre emanating from sisal prepared with $ZnCl_2$ and KOH provided micropores with a suitable width for the adsorption of PFOA and PFOS. In this study, ACF from sisal produced a micropore- material with a distribution pore of about 8 Å. This was in agreement with the previous report from by Chen *et al.* (2005), who achieved an ACF with a similar pore diameter.

D. Removal of PFOA and PFOS from drinking water using Agave sisalana activated carbon fibre

In this section, experimental data proved that PFOA and PFOS were removed from polluted water using ACF produced from *Agave sisalana* treated with KOH in an electro-physico-chemical system. Samples 1 ($ZnCl_2$ – 1.59 M) and 2 (KOH – 0.54 M) were used. Both sorbents showed asignificant pollutant removal.

TABLE IV. REMOVAL OF PFOA AND PFOS FROM TAP POLLUTED WATER WITH ACF

Sample Name	PFOA (ng/L)	PFOS (ng/L)	Time (min)	% removal	
	Calc. Conc.	Calc. Conc.		PFOA	PFOS
KFP0	106.141	100.145	0	0	0
KF1	92.839	77.365	20	12.532	22.746
KF2	76.391	61.523	40	28.027	38.565
KF3	72.185	52.785	60	31.990	47.290
KP4	72.090	51.891	80	32.080	48.183
KF5	71.110	49.070	100	33.004	51.000
KF6	70.388	47.126	120	33.684	52.941

IV. CONCLUSION

The manufacture of activated carbon by chemical activation requires the use of chemicals. Samples prepared with KOH present the best percentage removal for PFOA and PFOS, where the satisfactory concentration for KOH was 0.54 M. This study demonstrated that an activated carbon fibre-based *Agave sisalana* successfully removed PFOA and PFOS from polluted tap water.

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Serge Imwer Mapan graduated with a Masters of Technology degree in Chemical Engineering cum laude at the Cape Peninsula University of Technology in 2015 under the tutelage of Profs SKO Ntwampe and MS Sheldon including Dr S Odisitse. He was part of the development of the perfluoroalkyl contaminants in the Bioreseorce Engineering Research Group.