EFFECTIVE REMOVAL OF EMERGING CONTAMINANTS FROM DRINKING WATER USING POWDERED ACTIVATED CARBON: A COMPARATIVE STUDY

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Abstract - The widespread and adverse health-related and environmental impacts of different types of emerging contaminants in water resources have become a global issue of concern. On the other hand, the ever increasing socioeconomic awareness, scientific knowledge, ecological apprehensions and health-related problems, people are more concerned about the widespread of emerging contaminants in water resources globally. Regulatory frameworks and toxicological studies require these compounds to be removed from water prior utilization by living organisms including humans. In this study, the effectiveness of different powder activated carbon on the removal of emerging contaminants (ECs) from drinking water was comprehensively evaluated. Real raw water samples were collected from the river that feeds the 12 ML/d drinking water treatment plant. Optimization studies was done using jar stirrers and specifically the one factor at a time. Effect of dosage was assessed as a preliminary appraisal. High-pressure liquid chromatography-mass spectrometry (HPLC-MS) was used to determine the levels of ECs. Assessed ECs comprise Sulfamethoxazole, Carbamazepine, 1.7 Dimethylxanthine, Tramadol, and Venlafaxine. Findings from this study successfully confirmed the removal of selected emerging contaminants from drinking water. The obtained results displayed a better performance when the powder activated carbon dosage was increased between 20 – 100 mg/L. A100% removal efficiency was achieved for Sulfamethoxazole at 20 mg/L; Carbamazepine at 40 – 60 mg/L;Venlafaxineat 40 – 80 mg/L and Tramadolat 40 – 100 mg/L.During comparisons of the two activated carbons, obtained results showed that the Sulfamethoxazole was removed on both occasion at the same dosage, i.e., 20 mg/L, followed by Carbamazepine in different dosage points of 40 mg/L (ARG PAC) and 60 mg/L (Conventional PAC), then Venlafaxine in different dosage points of 40 mg/L (ARG PAC) and 80 mg/L (Conventional PAC), Tramadol in different dosage points of 40 mg/L (ARG PAC) and 100 mg/L (Conventional PAC) and between 90 – 97% removal efficiency of 1.7 Dimethylxanthine was achieved at dosage points of 100 mg/L on both occasions (ARG PAC and Conventional PAC).Overall, the ARG PAC (commercial PAC) performed efficiently as compared to PAC (Conventional PAC) used at the water treatment plant. From the performed jar test experiments, the obtained results showed that 1.7 Dimethylxanthine was not completely removed but it reported ≥95% removal efficacyin 100 mg/L. However, with the increase in dosage, better removal was obtained. This might be due to the nature of the physical and chemical properties of the 1.7 Dimethylxanthine and its affinity to PAC. Henceforth, the obtained results show that PAC can be used to completely remove selected emerging contaminants from drinking water. In light of the obtained results, it is imperative to optimise operational conditions such as contact time in order to obtain the most efficient and effective removal of ECs from drinking water.

Keywords - Water Treatment Plant, Emerging Contaminants, Powder Activated Carbon, High-Pressure Liquid Chromatography-Mass Spectrometry, Adsorption

I. INTRODUCTION

Water treatment is a process of making water suitable for its application or returning its natural state. The treatment may include mechanical, physical, biological, and chemical methods. The objective of water treatment is to produce safe and potable drinking water. Drinking water should, ideally, be obtained from a raw water source free from pollution, however many previous studies have revealed the presence of emerging contaminants (ECs).Specifically, personal care products (PCPs), steroidal hormones, and pharmaceuticals are among the ECs that are discharged to environment and water resources through various sources such as industrial and domestic wastewater, runoff from concentrated aquaculture and animal feeding operations, landfill leachate, urban run-offs, and effluent discharge from wastewater treatment plants. ECs are often available in relatively small concentrations in water resources, however their occurrence as transformation products can still cause detrimental effect on animal and human health as well as terrestrial and aquatic life on exposure. Some of the reported human health effects of ECs includes damage to the kidney, liver, and nervous system, and possible cancer and carcinogenic risks. The PPCPs, in aquatic bodies, affect water quality, ultimately affecting drinking water supply, human health and the ecosystem. The PPCPs group consists of food supplements, analgesics, antiseptics, beta blockers, sunscreen agents, diuretics, cosmetics, stimulant drugs, antibiotics, non-steroid, anti-inflammatory drugs (NSAIDs), antimicrobials, lipid regulators, fragrances, and several transformation products. A major concern caused by PPCPs is due to antibiotic residues, whose availability in water

resources can cause the occurrences of antibiotic resistance genes in bacteria. Due to their potential toxicity and negative impact on the human health and ecosystem, removal of ECs in the water resources has attracted the attention globally. Various technologies have been studied for effective removal of ECs. In the 20thcentury, water treatment methods progressed significantly with the incorporation of filtration processes in the treatment system and the introduction of chemical dosage methods. However, previous studies have shown that standard water treatment plant unit processes as displayed on figure 1, removes minute amount of theECs, suggesting that it is not an effective removal method.

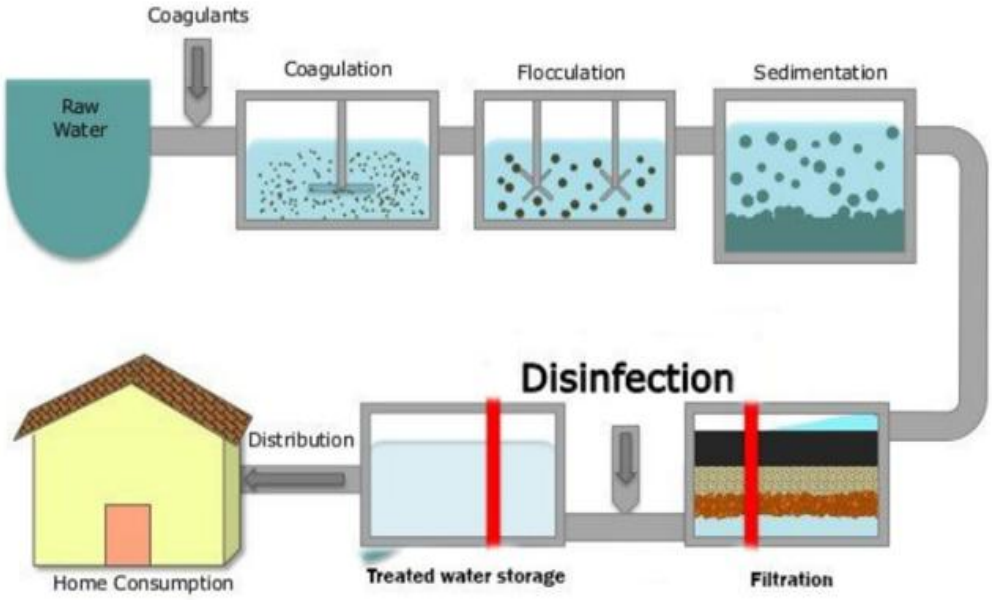

Figure 1: The typical sample unit processes within the drinking water treatment plant.

Other several technologies such as chlorination, ozonation,electrocoagulation,electro fenton process, photocatalysis etc. have also been studied however the Powdered Activation Carbon (PAC) showed better removal efficiency of the ECs. To date, many individual case studies have been published in peer-reviewed journals related to the removal of ECs by adsorption. The powder activated carbon (PAC) has been globally employed as an advanced physical technique due to itshigh specific surface area and porosity,reliability, and capability to remove up to 99.9 % of pollutants from aqueous solutions. The effective removal of the ECs using PAC depends on solute and adsorbent physicochemical properties. The key mechanism of removal is hydrophobic interaction for most nonpolar ECs.

Depending on the quality of the raw water, the extent of pollution and the regulations for safeguarding of public health, drinking water is treated by various methods before it reaches the consumer.Water to be supplied for public consumption must be fit for human consumption i.e meet the set drinking water standards. The PAC from different materials is effectively used to remove different ECs.In this study, the purpose is to employ the PAC as the absorbent for removal of ECs and compare their efficacy in a 12 mega liter drinking water treatment plant (WTP).

II. MATERIALS AND METHODS

2.1. Sampling location and sample collection

In this study, the real raw water sample in a 12 ML/d water treatment plant was collected inPretoria, South Africa. This river received water from tributary that passes through variety ofagricultural activities, industrial effluent, and treated sewage effluent, and these activities eventually introduce these ECs to the river that feeds the raw water source. The sampling was done according to standard methods (APHA, 2017) and permission was obtained from plant management prior to the collection of water samples. About 25 L sample was collected for preliminary optimization studies.

2.2. Chemical and reagents

The 1000 mg/L standards of different ECs compounds of different lot numbers were purchased from Merck - Supelco (SA) and used to prepare calibration standards and quality control standards respectively. The 100% HPLC grade Methanol and 20% Formic acid were purchased from Sigma Aldrich (SA). Analytical reagent (AR) grade chemicals were used and the water was purified by Milli-Q system (Millipore, Bedford, MA, USA). The 99.50% nitrogen gas and 99.99% argon gas were purchased from Air Liquide (SA).

2.3 Preparation of absorbent.

The 1000 mg/L stock solution was prepared by weighing approximately 1.000g of the analytical grade PAC and conventional PAC and diluting it up to the mark of each 1L volumetric flask using deionised water. The glassware used was of Class A to ensure that the volumes measured are correct.

2.4 Optimization of PAC using the jar test stirrer

The optimization experiments were conducted in a jar test apparatus (PHIPPS & BIRD Jar Tester).For each experiment, 1 L beaker was filled up to the mark with raw water. Various sets of jar tests were conducted for samples collected during June - October 2022 using two types of PAC. During the test, pre-determined dosages of PAC solution were added at 20, 40, 50, 60, 80 and 100 mg/L as per table 1. The fast-mixing speed was 400 rpm and continued for 2 min to dissolve the chemicals followed by a 40 revolutions per minute slow mixing speed for 8 min to promote floc formation enhancing particle collisions to larger flocs. Henceforward, the samples were left to be settled for 8 min and the supernatant was collected for determining the efficiency of ECs removal. The selected ECs of interest were Sulfamethoxazole (SMX), Carbamazepine (CMZ), 1.7 Dimethylxanthine (1.7 DXN), Tramadol (TMD) and Venlafaxine (VFX).

Sample Names	Dosages (mg/L)
PRWS 1	20
PRWS 2	40
PRWS 3	50
PRWS 4	60
PRWS 5	80
PRWS 6	100

Table 1: Dosages of the PAC used during jar test experiments

2.4.1 Emerging Contaminants removal efficiency calculation.

The removal efficiency of ECs was calculated using the following equation:

$$\text{Removal of ECs (\%)} = (N_{before} - N_{after}/N_{before}) \times 100 \quad (1)$$

Where, N_{before} is the concentration of the sample before PAC was addedand N_{after} is the concentration of ECs in the sample after the interaction with PAC.

2.5 Sample preparations

During sample preparations for ECs, sample extraction was done in triplicates. Thesolid-phase extraction (SPE) as shown in figure 2, was applied by using a vacuum manifold. About 200 mL of each water sample was filtered through filter paper to avoid sorbent clogging. In order to avoid the targeted analytes being lost during filtration, the pre-washed filter papers with 2 mL methanol used.

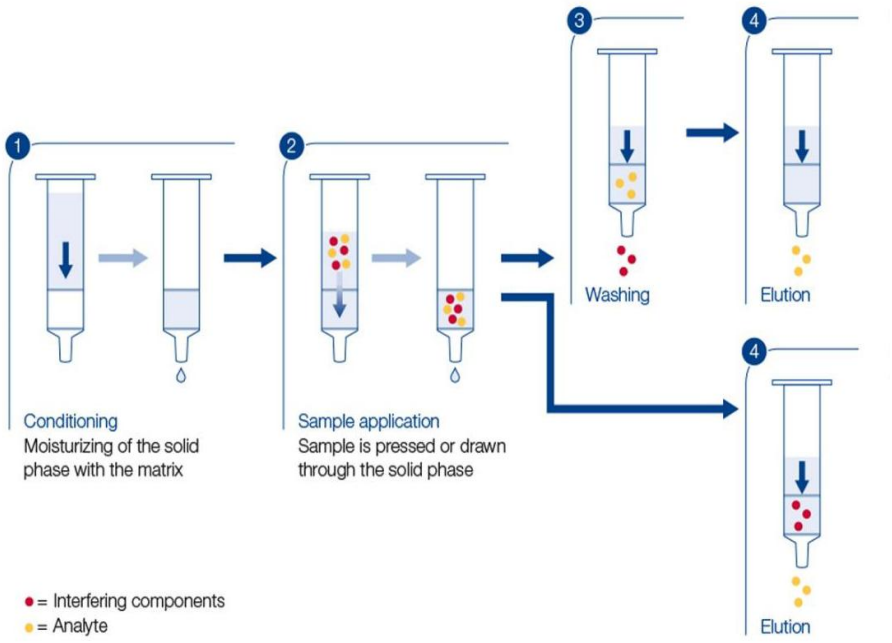

Figure 2: The typical sample preparation using the solid-phase extraction technique.

2.6 Characterization of ECs

Samples were analysed using an in-house HPLC-MS/MS method developed for a Water Research Commission of South Africa (WRC) project(K5/2733//3). The quality assurance sample (i.e., calibration, quality control and samples were in triplicate) were analysed to ensure the accuracy of the results. An ultra-performance liquid chromatograph (HPLC; Waters AQUITY) as shown on Figure 3, was used to acquire chromatography of target analytes.

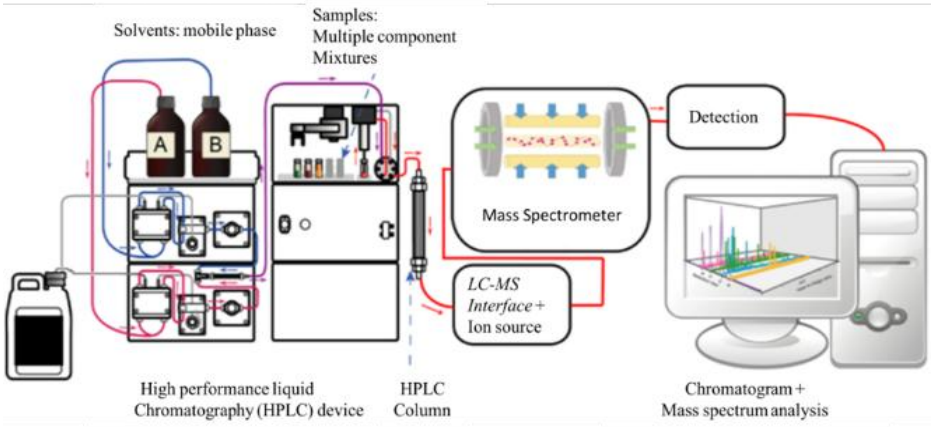

Figure 3: The typical components of the HPLC-MS.

Separation of the target analytes was achieved using de-ionised water spiked with0.1% formic acid (mobile phase-A), and 100% HPLC-grade methanol (mobile phase-B). The 100% mobile phase-A was used for starting conditions and maintained for 0.2 min, then reduced to 10% mobile phase-A over 6.8 min and finally to 0% mobile phase-A over 0.1 min (100% mobile phase-B). This was returned to 100% mobile phase-A over a period of 0.4 min and maintained for 2.5 min for re-equilibration. The total run time was 10 min. This method used a reversed-phase BEH C18 column (Waters AQUITY, 1.7 µm pore size, 2.1 x 100mm) equipped with a 0.2 µm in-line column filter. The columntemperature was maintained at 50 °C. The flow rate of the mobile phases was set at 0.4 mL/min with a sample injection volume of 2 µL. The HPLC was coupled with a triple quadrupole mass spectrometer (Xevo TQ-MS, Waters AQUITY) equipped with an electron spray ionisation source. All the analytes were determined using a positive ionisation mode (ESI+). Nitrogen was used as both nebulising and desolvation gas, and argon as collision gas. Acquisition of the HPLC-MS data was achieved using a multiple reaction monitoring (MRM) mode using two fragment ions for each compound where possible.Linearity of a reference standard calibration curve for each target analyte was assessed using a 10-point concentration range, from 1 µg/L to 750 µg/L, in the same solvent as the re-constituted water samples (MeOH). The integration of the analyte standard curves and surface water sample concentrations were determined using the TargetLynx software (Version 4.1, Waters).When a sample is injected, it is adsorbed on the stationary phase, and the solvent passes through the column to separate the compounds one by one, based on their relative affinity to the packing materials and the solvent.

III. RESULTS AND DISCUSSIONS

In this study, different dosages of PAC (20−100 mg/L)as shown in Table 1; [i.e AR Grade PAC (ARG PAC) and conventional PAC] were used to determine and compare the removal efficiency of ECs from raw water samples using the same operational conditions of the jar stirrer. During the study, it was observed that at the high PAC dosage, better percentage removal efficiency of ECs was achieved.

For ARG PAC, the obtained results as shown in Figure 4 (a). The resultsshows≈100% removal efficiency for SMXin20 mg/L;TMD, VFX and CMZin40 mg/L; followed by 95% for 1.7 DXN in 100 mg/L.

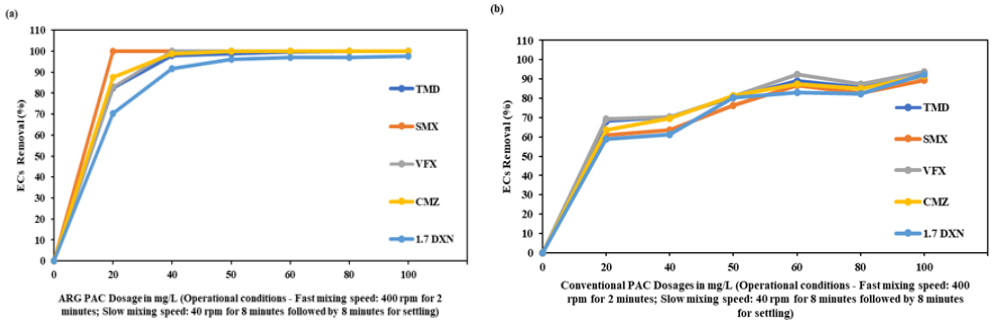

Figure 4: The obtained results of the selected ECs.

The results showed effective removal of ECs at low dosage for SMX, followed by the TMD, VFX and CMZ at double the amount of dosage whilst the better removal of 1.7 DXN was obtained at the highest dosing point. When conventional PAC was used, the obtained results as shown in Figure 4 (b) shows the 100% removal efficiency was achieved for SMX in 20 mg/L, CMZ at 60 mg/L, VFX at 80 mg/L followed by 90 – 97% for both TMD and 1.7 DXN achieved in 100 mg/L. When comparing the performance of the two types of activated carbon as per Figure 5 (a), it shows that the SMX was removed on both occasion at the same dosage of 20 mg/L, followed by CMZ in different dosage of 40 mg/L (ARG PAC) and 60 mg/L (Conventional PAC), then VFX in different dosage of 40 mg/L (ARG PAC) and 80 mg/L (Conventional PAC), TMD in different dosage of 40 mg/L (ARG PAC) and 100 mg/L (Conventional PAC) and between 90 – 97% removal efficiency of 1.7 DXN was achieved at dosage points of 100 mg/L on both occasions (ARG PAC and Conventional PAC).

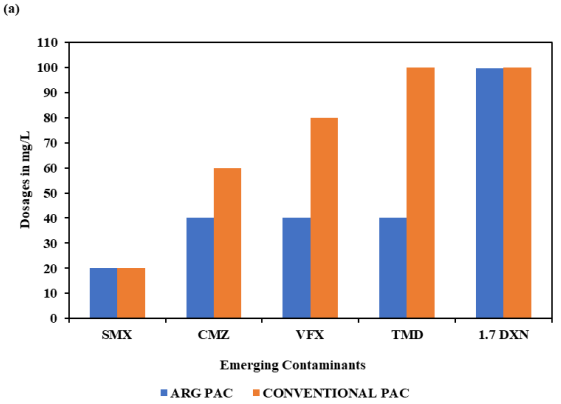

Figure 5: The comparisons of effective removal of ECs using ARG and Conventional PAC.

IV. CONCLUSION AND RECOMMENDATIONS

This study successfully demonstrated the effectiveness of PAC on the removal of selected ECs from authentic aqueous matrices. The two types of activated carbons used in this study, were derived from the different material and they comprise the analytical grade (ARG) and conventional PAC from coal. The ARG PAC was of higher purity when compared to the conventional PAC. When comparing the efficacy of the two types of PAC used, the ARG PAC showed more effective removal than the conventional PAC and this might be due to the different material source that were used to form those PAC. Obtained results revealed that when high PACdosage was used, better removal efficiencies for treating the selected ECs was achieved. On overall, ARG PAC performed efficiently than the conventional PAC which is currently used during water treatment process. Specifically, SMX was completely removed from raw water at 20 mg/L of dosage and 89% was achieved at 100 mg/L of dosage. The other compounds were completely removed from raw water sample as follows: CMZ from 40 – 60 mg/L and 92% at 100 mg/L; VFX from 40 – 80 mg/L and 94% at 100 mg/L; TMD from 40 – 100 mg/L and 91% at 100 mg/L. In all optimization experiments, the obtained results showed limited performance to 1.7 DXN, with the highest efficiency achieved for 1.7 DXN of ≥95% in 100 mg/L.

The ARG PAC is of high purity compared to the conventional PAC hence the results showed the highest removal efficiency when using ARG Grade. During the study, it was observed that, at the high PAC dosage, there was better removal efficiency of ECs hence denoting that the removal efficacy is dosage dependent. In conclusion, PAC can continuously be employed to remove the ECs but operating conditions such as contact time need to be optimized for effective and efficient performance. Where possible, the PAC of higher percentage purity must be employed for better removal efficacy.

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