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## **Removal of dissolved organic matter fractions from reverse osmosis concentrate:**

### **Comparing granular activated carbon and ion exchange resin adsorbents**

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#### **Abstract**

Reverse osmosis (RO) generates a concentrate (ROC) containing dangerous levels of pollutants including dissolved organic carbon (DOC). Adsorption experiments were conducted to study the effectiveness of removing DOC and its fractions from ROCs produced in a water reclamation plant using three adsorbents tested individually and in sequential combination. The ROCs had 23-42 mg/L DOC which contained 83-90% hydrophilics. These hydrophilics comprised 72-76% humics, 2-3% biopolymers, 5-7% building blocks, and 16-18% low molecular weight neutrals. Granular activated carbon (GAC) removed a larger amount of DOC

than two strong base anion exchange resins (Purolite A502PS, Purolite A860S). In both batch and column experiments, the adsorptive removal of the hydrophobic fraction was greater for GAC than for the Purolites. Humics present in hydrophilic fraction was completely removed by Purolites but only partially by GAC. In the sequential adsorption batch experiment, GAC followed by Purolite treatment removed more hydrophobics, however, Purolite followed by GAC removed more humics. Almost 100% of humics was removed for all doses of adsorbents when Purolite served as the first treatment. It is concluded that the order of adsorbent use for effectively treating ROC depends on the target DOC fraction intended to be removed.

**Keywords:** adsorption, granular activated carbon, LC-OCD DOC fractions, Purolite resins, reverse osmosis concentrate

## **Highlights**

- Adsorptive removal of DOC from ROC was higher with GAC than with Purolite resins.
- Hydrophilics had much higher concentration than hydrophobics in DOC.
- Of the hydrophilic fractions, humics had the highest concentration.
- GAC removed hydrophobics more than Purolites; opposite trend for humics removal.
- Combination of two adsorbents increased removal of DOC fractions than single ones.
- Order of adsorbent use in the combination changes DOC fractions removal efficiency.

## **1. Introduction**

Water recycling by reclaiming wastewater is an attractive option for alleviating the global water shortage problem. Reverse osmosis is a widespread and successful process that

produces high quality recycled water which can be used as potable and non-potable water. However, a major problem with this process is the generation of a reverse osmosis concentrate (ROC) which commonly contains high concentrations of pollutants originally present in the feed wastewater; it includes dissolved organic carbon (DOC) (Shanmuganathan et al. 2017; Umar et al. 2015). DOC, which is generated from the decomposition of plants and microorganisms, is a major pollutant of water bodies and seriously endangers the quality of drinking water. It causes colour, taste and odour problems, acts as a substrate for bacterial growth and reduces the effectiveness of water treatment processes (Ahmed et al. 2018; Korotta-Gamage and Sathasivan 2017; Matilainen et al. 2010). Furthermore, it is a major contributor to disinfectant by-product formation which can adversely affect human health (Fleck et al. 2004; Gonsior et al. 2014). DOC can be nearly 4 times more concentrated in ROC than in wastewater (Jamil et al. 2018), making it a serious threat to the environment if it is discharged to water bodies without any prior purification process. For this reason, it is essential that proper treatment techniques are applied to ROC before it is disposed to water.

Of the various methods of removing DOC, the adsorption process is very attractive because of its simplicity, cost-effectiveness, efficiency at low concentration, and minimal waste production. However, because DOC consists of different fractions which differ in molecular weight, hydrophobicity/hydrophilicity, chemical structure and functional groups, the adsorbents selected in the treatment process may have different levels of effectiveness in removing these fractions. Activated carbon (Johir et al. 2015; Shanmuganathan et al. 2015; 2017; Velten et al. 2011; Wang et al. 2013) and ion exchange resins (Ahmad et al. 2012; Cornelissen et al. 2008; Johir et al. 2015; Shanmuganathan et al. 2014) have been used to remove DOC from wastewater or ROC. In some of these studies, the removal efficiency of different DOC fractions measured using LC-OCD (Gibert et al. 2013; Huber et al. 2011; Velten et al. 2011), such as hydrophobic, hydrophilic, biopolymers (MW > 20,000), humics (MW approx. 1000), building blocks (MW 300-500) and low molecular weight (LMW) neutrals

(MW < 350) was investigated. However, very few analyses compared a variety of adsorbents in their relative effectiveness for removing the different DOC fractions. Cornelissen et al. (2008) compared the removal efficiency of different DOC fractions from a wastewater using many anion exchange resins and reported that the removal efficiency differed markedly among the resins (1-60%). The weak base anion exchange resin and a strong base anion exchange resin with the lowest water content had the lowest removal efficiency. In another study, Shanmuganathan et al. (2014) reported that granular activated carbon (GAC) removed a higher percentage of total DOC and most of the DOC fractions from a wastewater compared to an anion exchange resin (Purolite A502PS).

Because the adsorbents have different abilities to selectively remove the various DOC fractions the adsorbents can be combined to take advantage of their different properties and aim at completely removing all fractions of DOC from water. Effectiveness of such a combination of adsorbents in removing the various DOC fractions has not been tested previously. Therefore, the objectives of the study were to: firstly, compare the removal efficiency of DOC and its fractions from ROC using GAC and two Purolite anion exchange resins in column and batch experiments; secondly, model the adsorption of DOC and its fractions on GAC and the ion exchange resins; and thirdly, evaluate the removal efficiency of DOC and its fractions using a combined GAC and anion exchange resin treatment, one adsorbent followed by the other. The two adsorbents have different degrees of hydrophobicity and surface ionic properties and therefore are expected to differ in their efficiencies of removing the individual DOC fractions which have different chemical properties.

## **2. Materials and methods**

### **2.1. Reverse osmosis concentrate (ROC) and adsorbents**

Reverse osmosis concentrate (ROC) collected from an advanced water reclamation plant in Sydney, Australia was used in this study. The plant treated a combination of stormwater and biologically treated sewage effluent. The RO treatment resulted in approximately 72% of water being recovered, which generated a rejected concentrate (ROC) of about 28%. The characteristics of the ROC are summarised in Table 1. The physico-chemical characteristics of the adsorbents used in the study are presented in Table 2.

## **2.2. DOC analysis**

A liquid chromatography-organic carbon detection unit (LC-OCD) (DOC-Labor Dr. Huber, Germany) was used to measure the concentrations of the DOC fractions in the ROC. It is a size-exclusion chromatographic procedure combined with organic carbon detection facility. It separates the DOC fractions according to their molecular weights (MW). The four major fractions measured in this method are: biopolymers (>20,000 g/mol), humics (500-1200 g/mol), building blocks (weathering product of humics) (350-500 g/mol), and low molecular weight (LMW) neutrals (<350 g/mol) (Amy et al. 2011; Jamil et al. 2016; Shanmuganathan et al. 2015; Velten et al. 2011). More details of the procedure are presented elsewhere (Jamil et al. 2019).

**Table 1.** Characteristics of the ROC.

Parameter	Unit	Value
pH		7.5-8.2
Conductivity	mS/cm	2.2-3.5
DOC	mg/L	23-42
Mg <sup>+2</sup>	mg/L	50-70
Ca <sup>+2</sup>	mg/L	65-80
K <sup>+</sup>	mg/L	40-55
Na <sup>+</sup>	mg/L	350-400
Silica as Si	mg/L	1-6
PO <sub>4</sub> <sup>-3</sup>	mg/L	5-15
SO <sub>4</sub> <sup>-2</sup>	mg/L	155-170
NO <sub>3</sub> <sup>-</sup>	mg/L	20-35
Cl <sup>-</sup>	mg/L	420-530
F <sup>-</sup>	mg/L	1-3

**Table 2.** Characteristics of the adsorbents.

	PU-1	PU-2	GAC*
	Purolite A502PS*	Purolite A860S**	
Type and structure	Polystyrene cross-linked with divinylbenzene	Polyacrylic cross-linked with divinylbenzene	Coal based (MDW/4050CB)
Functional group	R-(CH <sub>3</sub> ) <sub>3</sub> N <sup>+</sup>	Quaternary ammonium	-
Ionic form (as shipped)	Cl <sup>-</sup>	Cl <sup>-</sup>	-
Exchange capacity (eq/L)	0.85	0.8	-
Moisture retention (%)	66-72	66-72	Maximum 2
Particle size (mm)	0.425 – 0.600	0.300 – 1.200	0.42-1.68
Surface area (m <sup>2</sup> /g)	21	–	Iodine number 1000, BET 1000

\*Shanmuganathan et al. (2014), \*\*Purolite ® (2017)

### 2.3. Batch adsorption experiments and adsorption data modelling

The first set of equilibrium batch adsorption experiments were conducted with each of the adsorbents, GAC, PU-1 and PU-2 individually, using a series of glass flasks containing 200 mL ROC and adsorbent doses of 0.1 - 5 g/L at room temperature ( $25 \pm 1$  °C). This was done by agitating the suspensions in a Ratek Platform Mixer at 120 rpm for 22 h. The suspensions were then filtered through a 0.45 um filter and the filtrates were analysed for DOC. Percentage adsorption of DOC was calculated using the following equation:



$$\text{Percentage adsorption (\%)} = \frac{(C_0 - C_e)}{C_0} \times 100$$

where,  $C_0$  is initial concentration of DOC (mg/L) in ROC, and  $C_e$  is the equilibrium concentration of DOC (mg/L).

Because GAC and Purolites were selective in their adsorption of the various DOC fractions, a second set of experiments were conducted to determine the percentage of the fractions removed when ROC was first treated with GAC and subsequently treated the remaining un-removed DOC by adsorption onto PU1 and vice-versa. The method used for the experiments was the same as that for individual adsorbents except the adsorbent doses were 0.7-5 g/L. The percentage adsorption of the DOC fractions was determined by a method similar to that of DOC adsorption explained in the first set of experiments.

The modelling was conducted by fitting the data to Langmuir (Ahmad et al. 2012) and Freundlich (Johir et al. 2015) adsorption isotherms which are very commonly used to describe the experimental data of adsorption isotherms. The equations describing these two models are as follows:

Langmuir model

$$q_e = \frac{Q_m b C_e}{1 + b C_e}$$

Freundlich model

$$q_e = K_f C_e^{1/n}$$

where,  $C_e$  = equilibrium concentration of DOC or its fractions (mg/L),  $q_e$  = amount of DOC or its fractions adsorbed per unit mass of adsorbent (mg/g),  $Q_m$  = maximum amount adsorbed per unit mass of adsorbent (mg/g),  $b$  = Langmuir affinity constant (L/mg),  $K_f$  = Freundlich constant (mg/g) (L/mg)<sup>1/n</sup> and  $n$  = another Freundlich constant.

## 2.4. Column adsorption experiment

Column experiments were conducted using an acrylic glass tube of internal diameter 2 cm containing each of the adsorbents packed to a bed height of 25 cm. A stainless-steel sieve was attached to the bottom of the tube to keep the adsorbents in place and a layer of glass beads was placed on top of the adsorbents bed in order to provide a uniform flow of solution through a fixed-bed column. ROC was passed through the column in the up-flow mode with a velocity of 40 mL/min (6.3 m/h). A peristaltic pump was utilised for pumping the ROC through the column. The effluent samples were collected periodically, filtered through a 0.45 µm filter, and the filtrate analysed for total DOC and its various fractions.

## **2.5. Column adsorption capacity and partition coefficient calculations**

Adsorption capacities of the DOC and its fractions in the column experiments were calculated using the following equation which is similar to the one used by Velten et al. (2011):

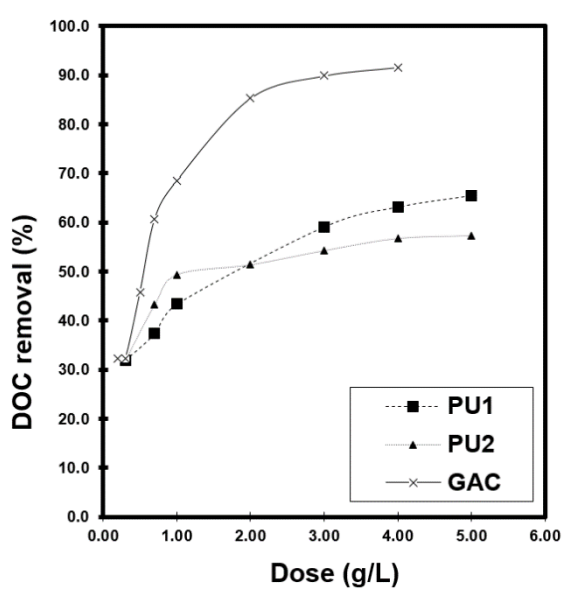
$$q = Q \times \Delta t / m [(C_o - 0.5 (C_{out,t} + C_{out,t-1}))]$$

where,  $q$  is adsorption capacity between two consequent times (mg/g adsorbent),  $Q$  is flow rate (L/min),  $\Delta t$  is time interval between the two sampling times (min),  $m$  is mass of adsorbent in column (g),  $C_o$  is influent concentration (mg/L),  $C_{out,t}$  is effluent concentration at time  $t$  (current sampling time (mg/L)), and  $C_{out,t-1}$  is effluent concentration at time  $t-1$  (previous sampling time) (mg/L). The partition coefficient,  $k_p$  was calculated by dividing  $q$  by  $C_{out,t}$  for each time period.  $k_p$  is the ratio of the amount adsorbed to the equilibrium concentration in solution, which is a measure of the absorbability of the DOC and its component on the adsorbents (Velten et al. 2011).

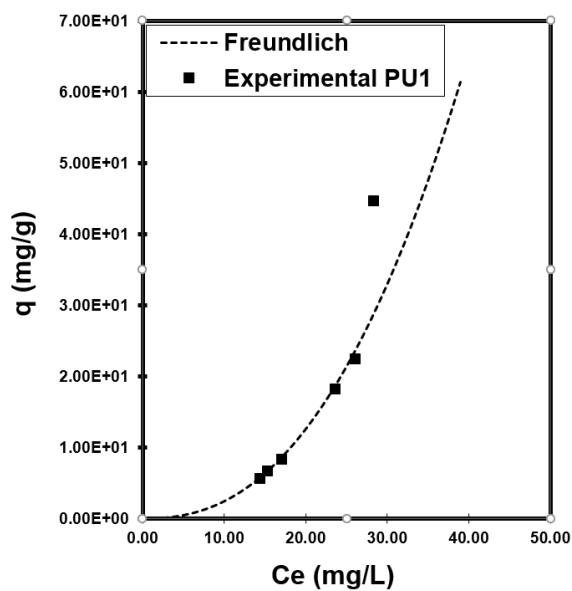
## **3. Results and discussion**

### **3.1. Batch adsorption of DOC on individual adsorbents**

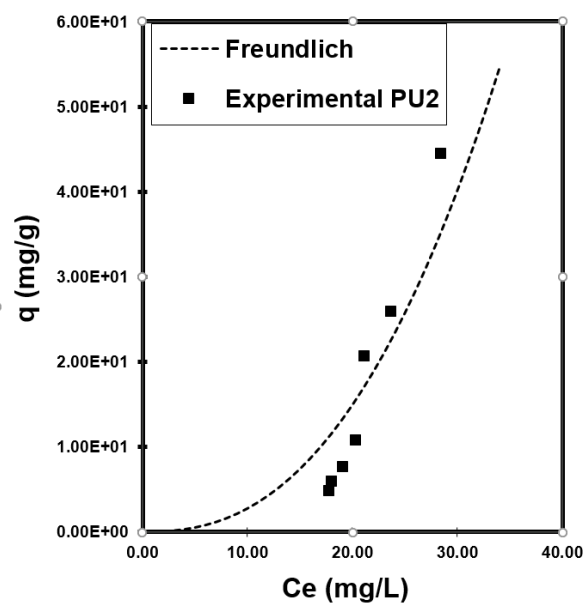
Of the three adsorbents, GAC removed a higher percentage of DOC than the two Purolite ion exchange resins which had similar removal percentages (Fig. 1a). The adsorption isotherms for the Purolite resins showed S-type curves, thus indicating that DOC adsorption was weak at low concentration because of the polar nature of these adsorbents where the hydrophobic DOC fractions have low adsorption affinity (Fig. 1 b,c). In contrast, GAC exhibited an L-shaped curve (Fig. 1d). These results agree with those of previous studies where an S-type isotherm was obtained on Purolite A502PS resin (Shanmuganathan et al. 2014) and L-type curve on GAC (Shanmuganathan et al. 2015) for the adsorption of DOC.



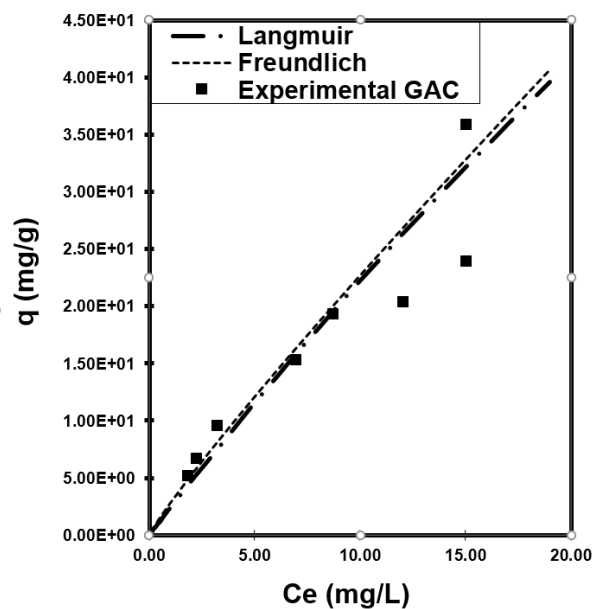
(a)



(b)



(c)



(d)

**Fig. 1.** (a) Percentage removal of DOC (initial concentration 41.8 mg/L) by GAC, PU1 and PU2; and adsorption isotherms of (b) Purolite-PU1 (c) Purolite-PU2 and (d) GAC.

**Table 3.** Langmuir and Freundlich model parameters for the adsorption of DOC.

Adsorbents	Langmuir			Freundlich		
	$q_m$ (mg/g)	$b$ (L/mg)	$R^2$	$K_f$ (mg/g(L/mg) <sup>1/n</sup> )	$n$	$R^2$
GAC	300	0.01	0.88	2.80	1.10	0.88
Purolite (PU1)	-	-	-	0.01	0.42	0.97
Purolite (PU2)	-	-	-	0.01	0.41	0.97

Adsorption of DOC fitted well to the Langmuir model only for GAC resulting in a maximum adsorption capacity of 300 mg/g (Table 3). However, the Freundlich model fitted well to the adsorption data of all three adsorbents. The Freundlich  $K_f$  parameter, which reflects the adsorption capacity, was much higher for GAC than the Purolite resins agreeing with the data on percentage removal of DOC (Fig. 1a). The Freundlich parameter,  $1/n$  was between 0.1 and 1, indicating a favourable adsorption for GAC and suggesting there was a stronger interaction between the GAC and DOC (Sarkar et al. 2010; Yakout et al. 2013). The  $1/n$  values for DOC adsorption on Purolite resins were greater than 1 indicating unfavourable adsorption. Shanmuganathan et al. (2014) also reported  $1/n$  value higher than 1 (1.55) for DOC adsorption on Purolite A502PS resin from wastewater RO feed and suggested an unfavourable adsorption process. In contrast,  $1/n$  values for DOC adsorption on activated carbon have been reported to be less than 1, therefore indicating favourable adsorption (Gui et al. 2018; Treguer et al. 2006).

### 3.2. Column adsorption of DOC fractions on individual adsorbents

The ROC used in the experiments with the three adsorbents had different DOC concentrations but the percentages of the different DOC fractions are nearly the same (Table 4). The concentration of the hydrophilic fraction is 5-9 times that of the hydrophobic fraction. Among the hydrophilic fractions, humics constituted the predominant fraction followed by the LMW neutrals. The same order of the DOC fraction concentrations was reported for another ROC by Shanmuganathan et al. (2015).

**Table 4.** DOC fractions concentrations (mg/L) in ROC used in column experiments with the three adsorbents

Adsorbents	DOC	Hydropho- bics*	Hydrophi- lics*	Biopoly- mers**	Humics**	Building blocks**	LMW neutrals**
GAC	22.69	2.19 (10)	20.5 (90)	0.66 (3)	15.51 (76)	0.99 (5)	3.35 (16)
PU1	36.11	4.21 (12)	31.90 (88)	0.67 (2)	23.00 (72)	2.17 (7)	5.81 (18)
PU2	41.90	7.40 (18)	34.60 (83)	0.71 (2)	26.00 (75)	2.10 (6)	6.21 (18)

\*Number with parentheses is % of DOC; \*\*Number within parentheses is % of hydrophilics

The breakthrough curves for the different DOC fractions in the three adsorbents are presented in Fig. 2. The breakthrough for the hydrophobic fraction was higher for the two Purolites (higher Ct/Co) compared to that in GAC. This shows that the adsorption of hydrophobics was higher in GAC than in Purolites, which was probably due to the hydrophobic character of GAC (Nam et al. 2014). The ionic Purolites had lower adsorption capacities than GAC as evidenced by the higher breakthroughs. A striking feature of the breakthrough curves is that the humics fraction which is the major hydrophilic fraction in DOC (Table 4) was completely removed by the Purolites

but GAC removed noticeably less. This may be due to the ionic bonding of the negatively charged humics with the positively charged Purolites.

The amounts of the different DOC fractions adsorbed are mainly governed by the relative concentrations of the fractions in the ROC. In this respect, humics which had the highest concentration also had the highest adsorption capacity in all three adsorbents, despite the adsorption capacity in Purolites being higher than in GAC for the reasons explained previously under breakthrough curves (Fig. 3). Biopolymers, LMW neutrals and building blocks revealed low adsorption capacities in all adsorbents because of their low concentrations in ROC.

The partition coefficient which determines the absorbability of the DOC fractions (amount adsorbed/concentration in solution) (Velten et al. 2011) was different between the Purolites and GAC for DOC, hydrophobics and humics (Fig. 4). Consistent with the breakthrough curves (Fig. 2) and DOC removal percentages (Fig. 1), GAC due to its hydrophobic nature had higher partition coefficients than Purolites for DOC and hydrophobics. However, Purolites had much higher partition coefficients compared to GAC for humics because of their similar degree of hydrophilicity and ionic characteristics (oppositely charged Purolite surface and humics functional groups). The partition coefficient was extremely high for Purolites because the solution concentrations of humics were below the quantifiable value. Therefore, the lowest measurable solution concentration (0.001 mg/L) was used in the calculation of partition coefficients.

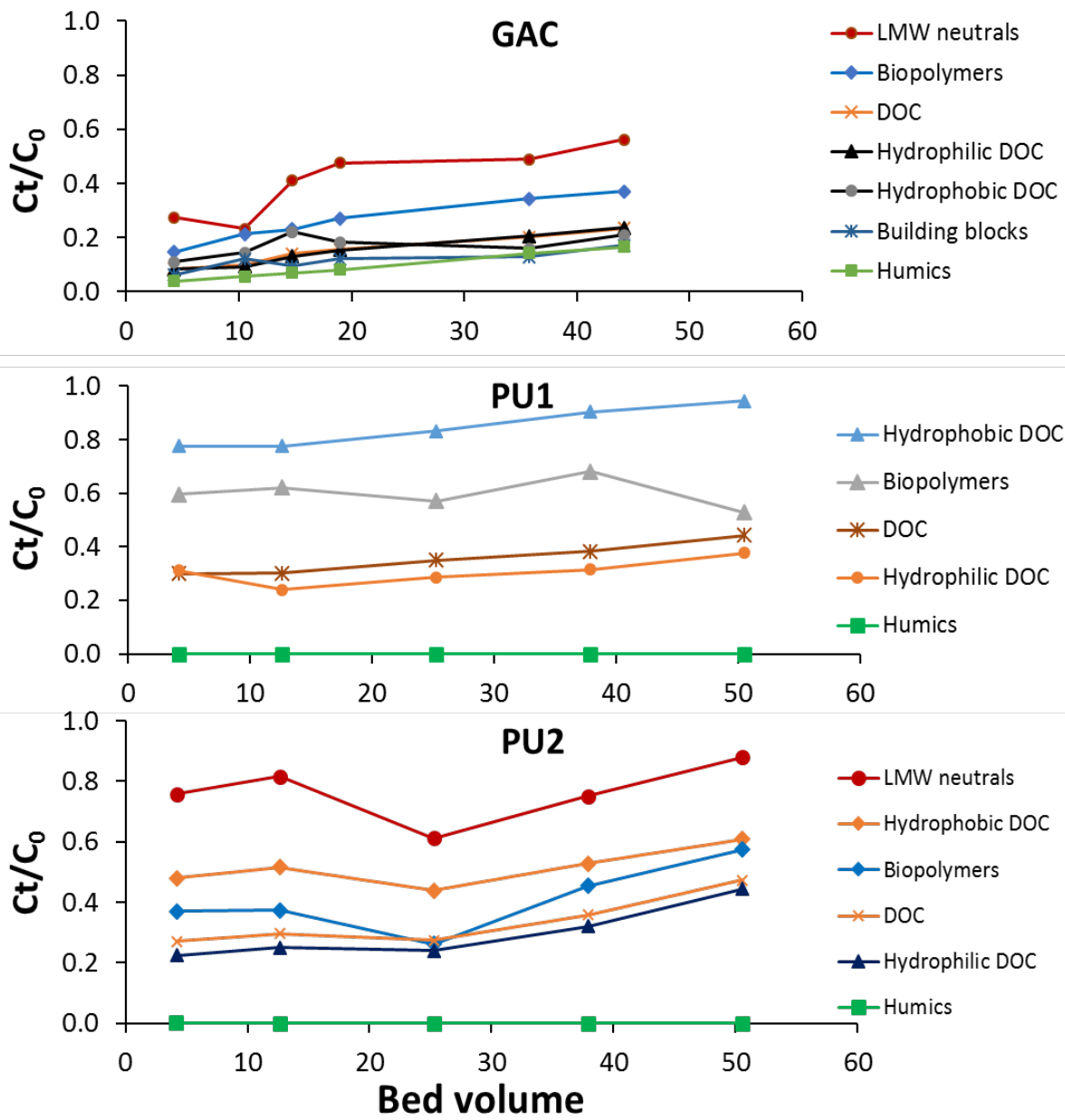


Fig. 2. DOC and its fractions' breakthrough curves for GAC, PU1 and PU2.



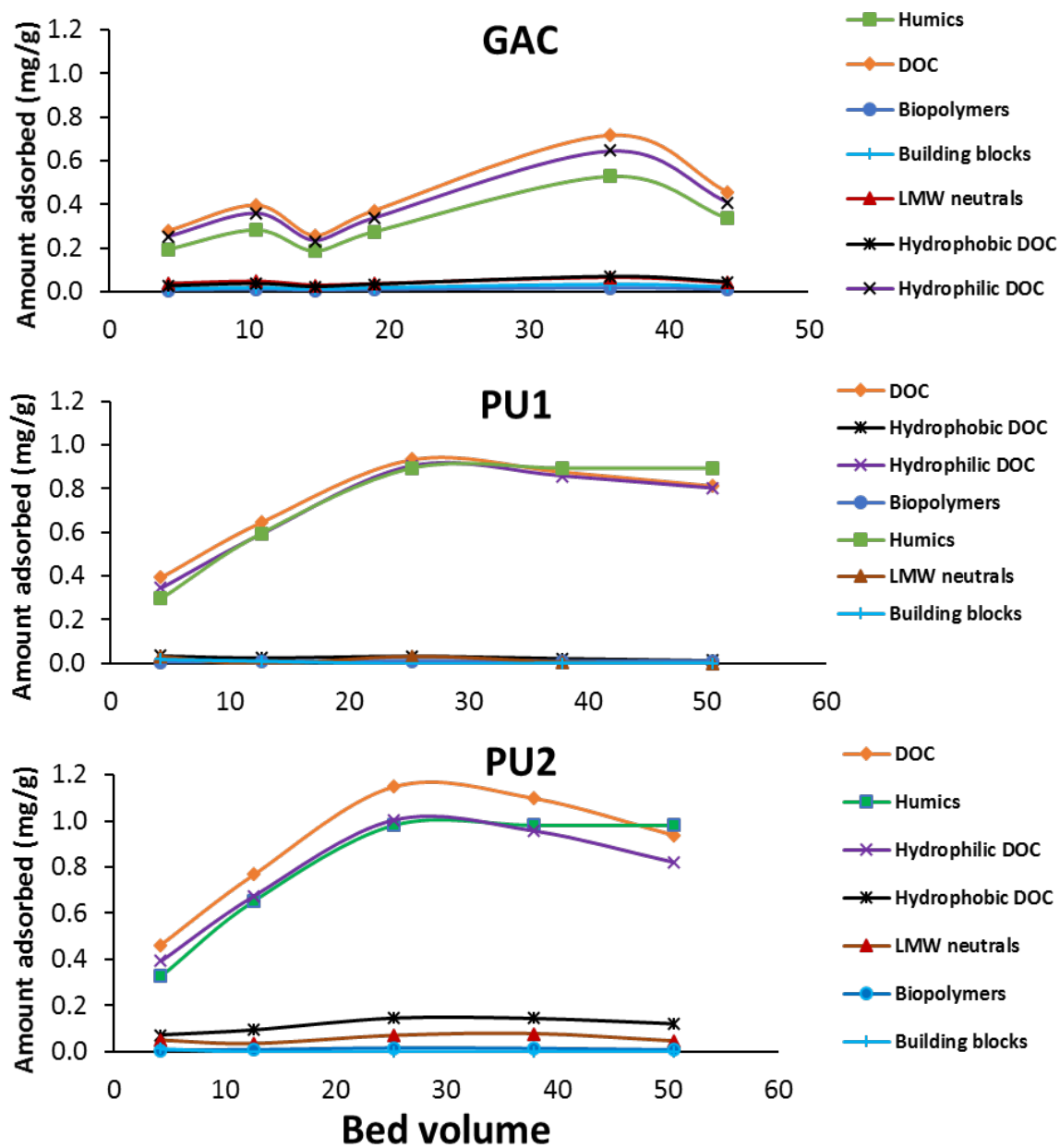


Fig. 3. DOC and its fractions' adsorption by GAC, PU1 and PU2.

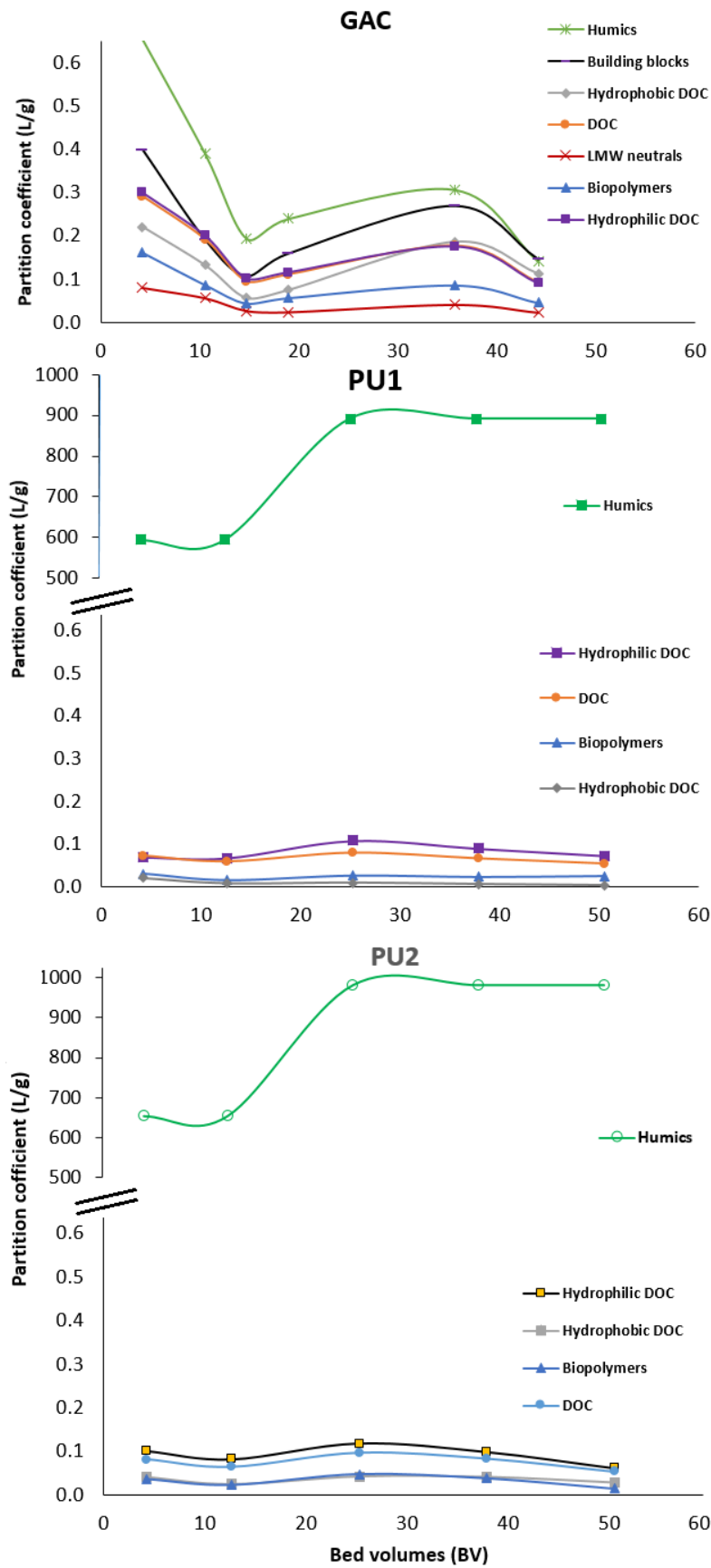


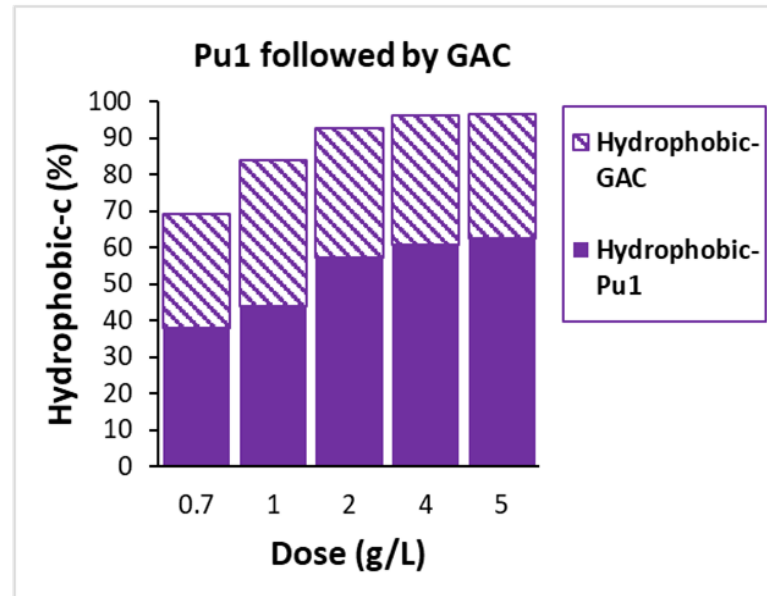
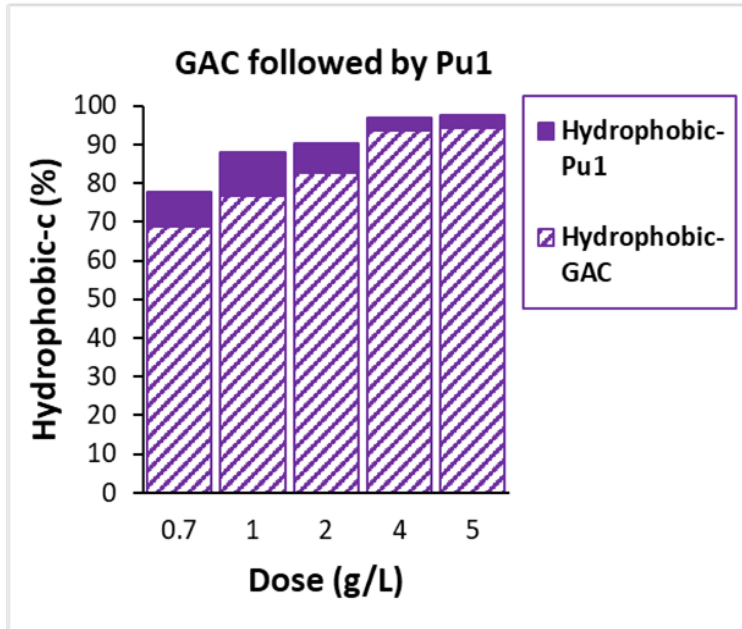
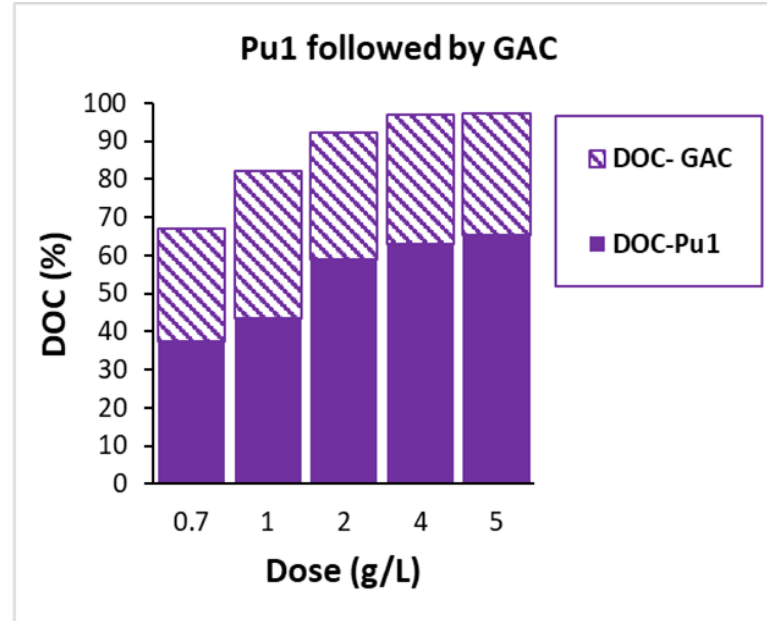
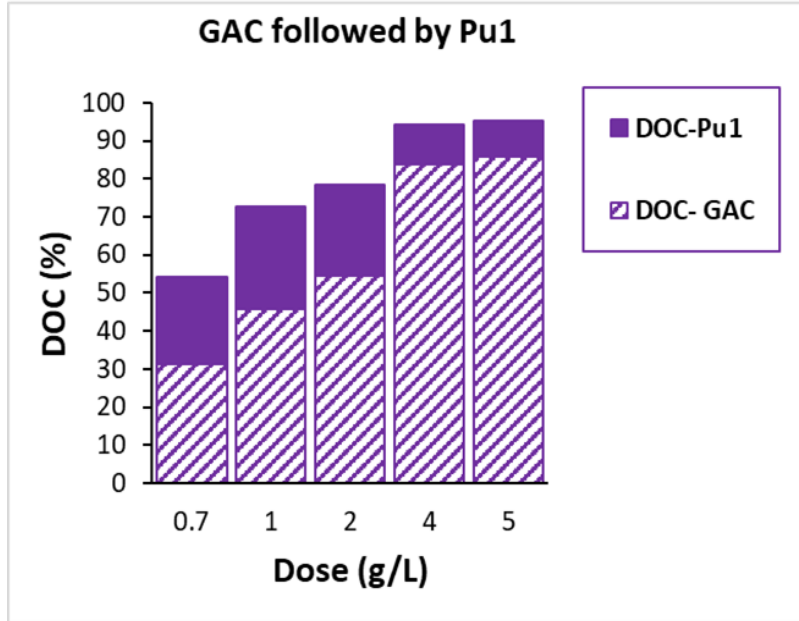
Fig. 4. DOC and its fractions' partition coefficients in GAC, PU1 and PU2.

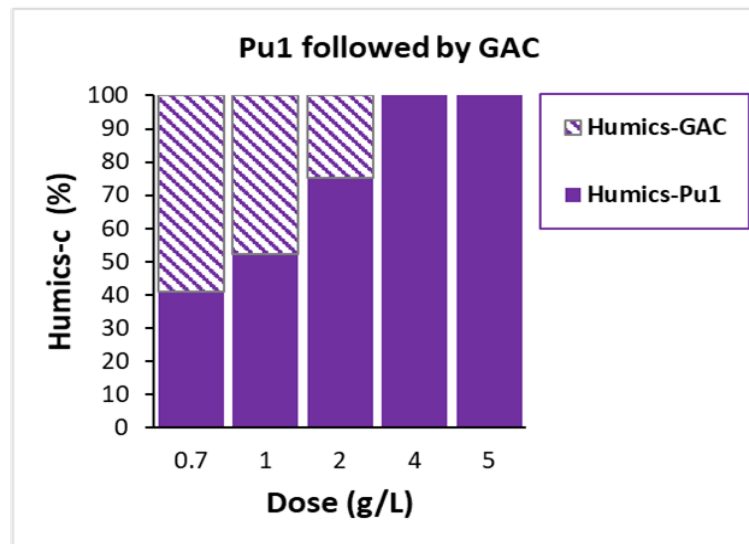
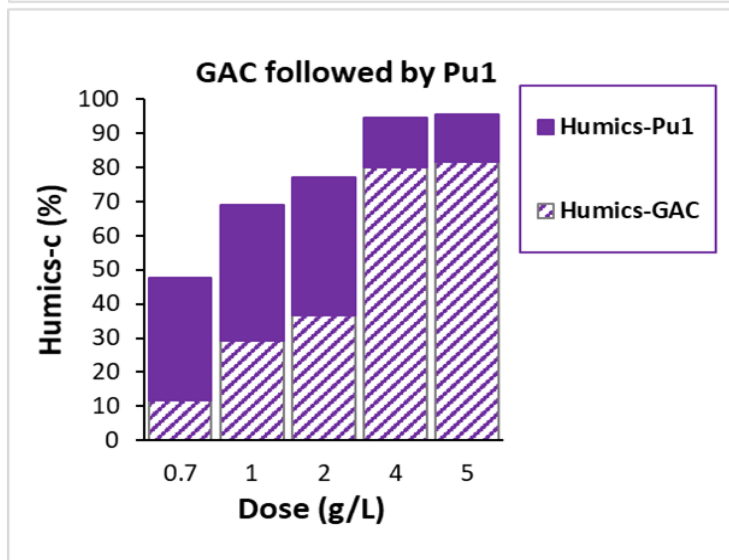
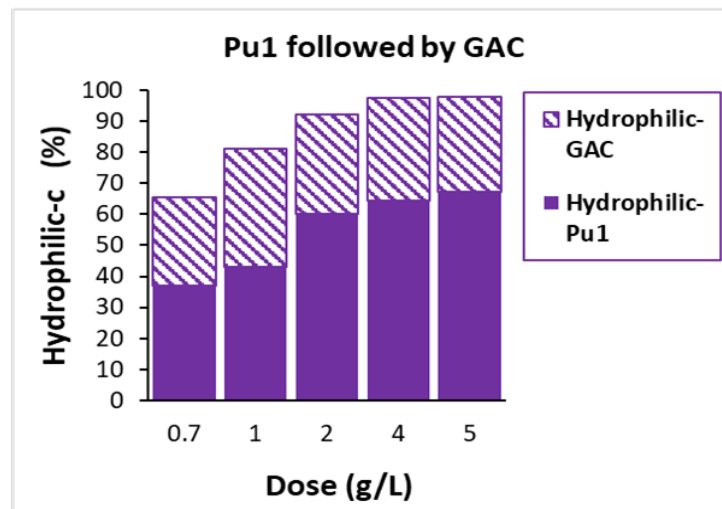
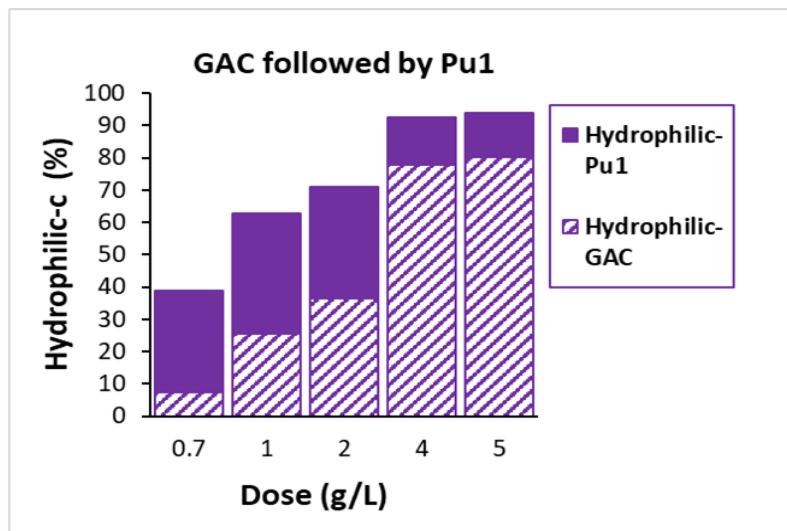
### 3.3. Batch adsorptive removal of DOC fractions by combined GAC and PU-1 adsorbents

In the experiment where the ROC was treated by GAC and PU-1, one after the other, at high doses, both adsorbents together removed 100% of all fractions regardless of the order of the treatments (Fig. 5). This is because there were many adsorption sites available for adsorption of the DOC fractions at high doses, and consequently there was very little competition for adsorption. At low doses, where the number of adsorption sites is limited, however, there were differences in the percentage removal between the two orders of treatments. GAC followed by Purolite treatment removed more hydrophobics but Purolite followed by GAC removed more humics. This is in accordance with the higher affinity of GAC for hydrophobics and Purolite for humics. Almost 100% humics were removed for all doses of adsorbents when Purolite was used as the first treatment. GAC removed a higher percentage of hydrophobics than Purolite and Purolite removed a larger percentage of humics than GAC when these adsorbents were used as the first adsorbent. Purolite followed by GAC treatment is a slightly better treatment than GAC followed by Purolite in removing the hydrophilic fraction (Fig. 5) may be because of the higher affinity of Purolite for hydrophilics as discussed in previous sections. Probably because of the larger percentage of hydrophobics removed, GAC was able to remove more total DOC, which replicated the individual adsorbent and column experimental results (Fig. 1 and 2).

Modelling of the first adsorbent treatment data of the combined adsorbent' sequential adsorption experiment covering all adsorbent doses of GAC and Purolite confirmed the findings derived from the individual dose results. The adsorption data for all fractions in the first treatment were satisfactorily described by the Freundlich model ( $R^2 = 0.77 - 0.99$ ) except the data for the hydrophilic fraction on GAC ( $R^2 = 0.62$ ) (Table 5). The data did not satisfactorily fit to the Langmuir adsorption model for any of the DOC fractions for both adsorbents which indicates that the adsorption process was not homogeneous and no monolayer formation of adsorbate on the adsorbent. The Freundlich  $K_f$  value which is related to the adsorption capacity was higher for the adsorption of hydrophobics and DOC on GAC than on Purolite, however, it was higher for the adsorption of humics on Purolite than on GAC. Nam et al. (2014) reported a positive correlation

between hydrophobicity of organic micropollutants and Freundlich  $K_f$  values obtained for the adsorption of the micropollutants on GAC and concluded that hydrophobic interaction of the micropollutants and GAC was the primary mechanism of adsorption of hydrophobic compounds. The Freundlich  $n$ -values which represent a measure of the intensity of adsorption also had the same trends as the  $K_f$ -values for the DOC and its fractions.





**Fig. 5.** Removal percentages of DOC and its fractions after adsorption on GAC followed by PU1 (Left-hand side) and on PU1 followed by GAC (Right-hand side).

**Table 5.** Freundlich model parameter values for the adsorption of DOC and its fractions on GAC and Purolite when they served as the first adsorbent treatment in the combined adsorbents treatments

Adsorbent	DOC/DOC fraction	Freundlich model parameters		R <sup>2</sup>
		$K_f$ (mg/g(L/mg) <sup>1/n</sup> )	n	
GAC	DOC			0.9845***
		1.126	0.952	
	Hydrophobic	3.420	0.730	0.9868***
	Hydrophilic	2.370	2.466	0.6246 (NS)
	Humics	1.664	1.757	0.9609***
PU1	DOC	0.010	0.422	0.9993***
	Hydrophobic	0.011	0.342	0.9963***
	Hydrophilic	0.042	0.480	0.9998***
	Humics	4.032	2.748	0.7731*

NS: not statistically significant, \*: significant (probability level = 0.05), \*\*\*very highly significant (probability level = 0.001) (Little and Hills 1978)

#### 4. Conclusions

Of the three adsorbents tested, GAC had a higher capacity than the two Purolite anion exchange resins in removing total DOC from ROCs. Freundlich constant related to the adsorption capacity was higher for GAC than that for the Purolites. GAC removed a higher percentage of the hydrophobic fraction of DOC than Purolites but the latter removed more humic fraction than the former.

In a sequential order of the adsorption process using two adsorbents, GAC followed by Purolite treatment removed more hydrophobics, but Purolite followed by GAC removed more humics. Almost 100% humics was removed for all doses of adsorbents

when Purolite served as the first treatment. GAC removed a higher percentage of hydrophobics than Purolite and Purolite removed a higher percentage of humics than GAC when these adsorbents were used first in the sequence. The adsorption of these DOC fractions was satisfactorily explained by the Freundlich model and the model parameter values agreed with the DOC fractions percentage removal data. The study demonstrated that: firstly, a combination of adsorbents would increase the removal percentage of all the different DOC fractions from ROC; and secondly, the order of adsorbent used in this combination can be manipulated to target the effective removal of the DOC fraction of interest.

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### **Notes**

The authors declare no competing financial interests

**Declarations of interest:** None

### **Authors' contributions**

Project supervision: Vigneswaran, Loganathan

Conceptualization and design of experiments: Vigneswaran, Loganathan, Jamil



Data collection and illustrations preparation: Jamil, Loganathan

Modelling: Kandasamy

LC-OCD analysis: Khourshed, Jamil

Materials and methodology (ROC): Listowski

Paper writing: All authors

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