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Effect of wood particle size on uptake and desorption study of chlorophenols by woody materials

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The impact of particle size on biosorbent performance is not well researched. Batch tests found pine and hardwood (HW) had a chlorophenol (CP) uptake of 3.1–7.1 mg g⁻¹ with an initial rapid uptake but equilibrium within 72 h. Pine particle size (0.6–4.75 mm) was relatively independent of surface area and equilibrium CP uptake. This was due to the elongate nature of the ground particles which had a length to width ratio of between 4:1 and 8:1 and a sieved particle size closely aligned with particle width rather than particle length. Intra-particle diffusion was a dominant sorption mechanism. Sorption–desorption isotherms of CP on pine and HW can be best described by the Freundlich equation. Desorption of CP showed greater hysteresis with increased hydrophobicity.

Keywords: chlorophenol; sorption–desorption; particle size; pine; hardwood

Introduction

Due to the widespread and pervasive nature of chlorophenol (CP) contamination of water, simple, effective treatment methods are needed. Currently, sorption is the most widely used method for CP removal. Studies have shown that sorption with granular activated carbon is an effective process both in potable and wastewater treatments.[1] Traditional sorbents (e.g. activated carbon and peat, inorganic materials) are expensive, particularly for a high-volume application (e.g. groundwater barriers). While many different low-cost sorbents have been studied,[2] there have been relatively few studies using widely available woody materials with organic contaminants.[3] There is a particular need to understand the behaviour of organic mulch (i.e. woody materials) for ‘biowall’ applications involving treatment through both sorption and as electron donor sources.[4] Of particular importance when designing large-scale filtration systems is the dependence of sorption on particle size. Smaller particle sizes require greater pre-processing and therefore increased sorbent cost. A better understanding of the impact of sorbent particle size on organic contaminant uptake is required.

MacKay and Gschwend [5] found mono-aromatic hydrocarbon uptake to be independent of whether the sorbent was pre-soaked pine chips, sticks or shavings. This may seem counter-intuitive since it is often postulated that particle surface area increases with a decrease in the particle size for particles with minimal internal porosity. Their results contrast with Barrera-Garcia et al.,[6] who found a dependence of particle size with uptake. The authors attributed this dependence to non-equilibrium sorption. Brás et al. [7] also found a relationship between particle size and

pentachlorophenol on pine bark. While there are contradictory studies on the relationship between particle size and uptake, it is well established that uptake is related to hydrophobicity. Desorption studies using pesticides on wood found hysteresis were linked to hydrophobicity.[8] However, there is relatively few literature describing desorption from woody materials.

The objectives of this paper were to give a greater insight into the extent and nature of the effect of particle size on the sorption of hydrophobic contaminants in aqueous solution. This topic is a relatively under-researched topic and has contradictory studies in the published literature. The sorption of 2-chlorophenol (2-CP), 4-chlorophenol (4-CP) and 2,4-dichlorophenol (2,4-DCP) onto pine and hardwood (HW) was studied using both kinetic and equilibrium batch tests at different particle sizes. Desorption experiments were also carried out in order to understand sorption irreversibility. It is hoped that a better understanding of these fundamental relationships will lead to enhanced sorbent design and utilization.

Experimental methodology

Preparation and characterization of sorbent

The sorbents used in this study were HW (Hardwood Chip, Soilco Pty Ltd, Australia) and pine chips (Superior Pine Chip, Soilco Pty Ltd). Any bark in the wood samples was removed by sorting, and the remaining wood was grounded to the required particle size using a hammer mill (Model No. ADEB80N2, John Morris Pty. Ltd, Australia). Sorbents were sieved such that the 4.75 mm material passed through a 6.5 mm sieve but was retained over a 4.75 mm mesh sieve.

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The 2.36 mm material passed through a 4.75 mm sieve, but was retained over a 2.36 mm mesh sieve. The 1.18 mm material passed through a 2.36 mm sieve but was retained over a 1.18 mm mesh sieve. The 0.6 mm material passed through a 1.18 mm sieve, but was retained over a 0.6 mm mesh sieve. All weights reported in the paper are as dry matter. The sieved pine and HW were further prepared by mixing with 0.6 mm glass beads (Burwell Abrasive Blasting Equipment, Australia) and then leached using tap water in a continuous flow column until the UV absorbance and electrical conductivity were at steady-state conditions (168 h). They were then washed with distilled water and dried in an oven at 50°C for 48 h, then stored in airtight desiccators at room temperature until use.

Total carbon and nitrogen for all solid materials were determined in duplicate using a Truspec Carbon Nitrogen determinator. Ash content was measured using the method for Total Volatile Solids [9] at 550°C. The specific surface areas and pore characteristics of solid-phase media were measured using Brunauer-Emmett-Teller (BET/N₂) (Micromeritics Tristar). Measured pine and HW surface areas and pore volume data (Table 1) are similar to other reported values for wood [10,11] and the cation exchange capacity (CEC) is consistent for pine.[4] The physical properties of sub-samples of the wood chips were measured with a micrometer. Representative sub-samples comprised 521 particles (0.5 g), 191 particles (1.0 g), 111 particles (2.5 g), 69 particles (6.6 g) and 210 particles (1.0 g) for the pine (0.6–1.18, 1.18–2.36, 2.36–4.75 and 4.75–6.5 mm) and HW (0.6–1.18 mm) samples, respectively.

Chemicals

Analytical-grade 2-CP, 4-CP and 2,4-DCP (Sigma-Aldrich Chemical Company, 99%) were used. Individual

1000 mg L⁻¹ stock solutions of each CP were prepared with Milli-Q water.

Sorption and desorption

In the kinetics experiments, sorbents (2-CP and 4-CP, 1 g and 2,4-DCP, 0.75 g) were immersed in 40 mL of 50 mg L⁻¹ solution within a 75 mL bottle and mixed at 150 rpm. A small quantity of liquid was withdrawn from the bottles at different predetermined time intervals for analysis. Equilibrium-based sorption experiments were carried out by equilibrating sorbents (2-CP, 4-CP, 2 g and 2,4-DCP, 1.5 g) in 40 mL of solution with different initial concentrations (20–300 mg L⁻¹) and were placed into a series of 75 mL bottles. The bottles were kept in an orbital shaker in a dark condition and continuously mixed at 150 rpm. These concentrations and equilibrium contact times (72 h) were established from the preliminary investigations. Equilibrium conditions were assumed to occur when there was less than a 5% change in uptake over 24 h.

Again, batch sorption experiments consisted of equilibrating 2 g of sorbent for 2-CP, 4-CP and 1.5 g for 2,4-DCP with particle size 1.18 mm at a nominal CP concentration of 300 mg L⁻¹ of 40 mL solution in a 75 mL serum bottle. The bottles were shaken immediately in an orbital shaker in a dark condition at 150 rpm. The samples were analysed for CPs. Following equilibrium, the liquid and solid phases were separated by centrifugation and decantation and then 40 mL of Milli-Q water was added to the bottle. Desorption study was carried out by four sequential decant–refill steps. After equilibrium, 20 mL of solution was removed from the solution and replaced by 20 mL of Milli-Q water. Then the bottles were shaken at 150 rpm for each desorption cycle (72 h) and analysed for residual CP. The equilibrium solid-phase loading on the sorbent was calculated by the difference of total amount sorbed and the amount desorbed in each step per unit sorbent.

The initial and final solution pH were between 4.5 and 5.2. This is below the pK_a values [12] of 2-CP (8.3–8.6), 4-CP (9.1–9.4) and 2,4-DCP (7.5–8.1) and therefore reflects sorption and desorption of the non-ionized form of the solute. Control samples showed no loss of CP during the experimental period. To verify this, calculated values for the fraction of CP in the headspace for these experiments were found to be less than 0.115% based on published Henry's constants. All experiments were performed at temperature (22°C) and were carried out without adding any pH buffer. Experiment was undertaken in duplicate with the maximum deviation from the mean on any sample being less than 5%.

Analytical methods

CP concentrations were determined by mixing 1 mL of extracted solution with 3 mL of Milli-Q water in a glass vial and centrifuging at 3000 rpm for 10 min to remove

Table 1. Physico-chemical properties of the sorbents.

Sorbents	Pine	Pine	Pine	Pine	HW
Particle size (mm)	0.6	1.18	2.36	4.75	1.18
BET surface area (m ² g ⁻¹)	0.65	0.45	–	0.79	0.55
Pore width (nm)	11.1	10.0	–	15.6	12.0
Total carbon (%)	–	48.7	–	–	48.3
CEC (cmol kg ⁻¹)	–	6.2	–	–	3.5
Lignin (%)	–	23	–	–	13
Cellulose (%)	–	57	–	–	61
Hemicellulose (%)	–	14	–	–	14
Residue (%)	–	6	–	–	12
Ash (%)	–	0.3	–	–	0.2
Average particle length (mm)	4.8	7.2	11.5	20.3	7.9
Average particle width (mm)	0.61	1.33	2.49	4.87	1.23
Length/width ratio	7.9	5.4	4.6	4.2	6.4
Interquartile distance (mm)	3	4	7	6	4

particulates. The solutions were then analysed for residual CP concentration with a UV–visible spectrophotometer (Shimadzu, Model UV-1700, Japan) using quartz cells. Control samples were used to adjust background absorbance values to account for any colour leaching from the sorbents; however, extensive pre-treatment of sorbents minimized the influence of leaching, as described in the section on sorbent preparation section of the paper. Further details of the analysis method are elsewhere.[3]

Data analysis

The amount sorbed, q_t (mg g^{-1}), at time t , was calculated according to the following equation:

$$q_t = \frac{V(C_0 - C_t)}{W}, \quad (1)$$

where C_0 is the initial concentration (mg L^{-1}), C_t the residual concentration (mg L^{-1}) at time t ; V the volume of the solution (L) and W the mass of the sorbent (g).

Equilibrium sorption capacity, q_e (mg g^{-1}), was calculated by the following equation:

$$q_e = \frac{V(C_0 - C_e)}{W}, \quad (2)$$

where C_e (mg L^{-1}) is the equilibrium concentration in the liquid phase.

The equilibrium solid-phase loading on pine and HW was calculated after desorption by the following equation:

$$q_e = \frac{(q_0W - C_eV)}{W}, \quad (3)$$

where q_0 is the equilibrium solid-phase loading after the initial sorption (mg g^{-1}), C_e (mg L^{-1}) the equilibrium concentration in the liquid phase after desorption and q_e the equilibrium solid-phase loading after desorption (mg g^{-1}).

Differences between sorption and desorption behaviour were evaluated using several different hysteresis indexes. Cox et al. [13] proposed a hysteresis coefficient H expressed as the following equation:

$$H = \frac{n_d}{n} \times 100, \quad (4)$$

where n_d and n are the Freundlich exponents for desorption and adsorption, respectively.

Zhu and Selim [14] proposed a hysteresis index based on the difference in area between the adsorption and desorption isotherms by the following equation:

$$\lambda = \left(\frac{n+1}{n_d+1} - 1 \right) \times 100. \quad (5)$$

When desorption occurs at the same rate as sorption, there is no hysteresis and $H = 100$ and $\lambda = 0$. Positive hysteresis

occurs when the desorption rate is slower than sorption and is indicated by $H < 100$ and $\lambda > 0$.

Sorption isotherm data were described by the two most widely used models. The Langmuir model is expressed by the following equation:

$$q_e = \frac{Q \times b \times C_e}{(1 + bC_e)}, \quad (6)$$

and the Freundlich model is expressed by the following equation:

$$q_e = KC_e^n, \quad (7)$$

where C_e is the equilibrium concentration in the liquid phase (mg L^{-1}) and q_e the equilibrium sorption capacity of sorbent (mg g^{-1}). Parameter Q represents the maximum sorption capacity (mg g^{-1}) with monolayer coverage on the sorbent particle while b the Langmuir constant related to the free energy of sorption (L mg^{-1}). K is the sorption capacity constant and n the Freundlich constant for surface heterogeneity.

Sorption isotherm models were fitted against experimental data using nonlinear regression within a Microsoft Excel spreadsheet.[15] This approach was used rather than linearization of the equations since linearization may result in improperly weighted data points during the analysis [16] and limit the accuracy of the fit.[15] The mathematical models were optimized for best fit using sum of error squares (SSE). Parameter fits with the lowest SSE values were considered best-fit. Regression coefficient (r^2) were also calculated. The SSE was calculated [16] by the following equation:

$$\text{SSE} = \sqrt{\frac{\sum (q_{e,\text{exp}} - q_{e,\text{calc}})^2}{N}}, \quad (8)$$

where $q_{e,\text{exp}}$ and $q_{e,\text{calc}}$ are the sorption capacity (mg g^{-1}) obtained from experiment and calculation, respectively, and N the number of data points.

The intra-particle diffusion rate on sorption can be expressed by the following equation:

$$q_t = k_{\text{id}}t^{0.5} + c, \quad (9)$$

where k_{id} is the intra-particle diffusion rate constant, q_t the amount sorbed per unit sorbent at time t and c the constant.

Results and discussion

Sorption

The sorption equilibrium time was determined from the batch kinetic tests. The initial sorption of CPs was rapid followed by a slower uptake rate and final equilibrium reached within 72 h. MacKay and Gschwend [5] found relatively rapid initial uptake for mono-aromatic hydrocarbon sorption onto pine with up to 33.6 h required for equilibrium on pre-soaked particles.

Table 2. Best fit isotherm parameters for the sorbents.

Sorbent	Sorbate	Langmuir isotherm				Freundlich isotherm			
		Q (mg g ⁻¹)	b (L ⁻¹ mg)	r^2	SSE	K	n	r^2	SSE
Pine (0.6 mm)	2-CP	3.3	0.012	0.980	0.11	0.14	0.541	0.999	0.02
	4-CP	3.5	0.014	0.980	0.12	0.17	0.534	0.999	0.02
	2,4-DCP	6.9	0.015	0.973	0.25	0.29	0.577	0.998	0.08
Pine (1.18 mm)	2-CP	3.3	0.011	0.974	0.11	0.12	0.561	0.999	0.03
	4-CP	3.4	0.013	0.974	0.13	0.16	0.533	0.999	0.02
	2,4-DCP	6.8	0.015	0.972	0.25	0.28	0.579	0.996	0.09
Pine (2.36 mm)	2-CP	3.1	0.010	0.979	0.10	0.11	0.559	0.999	0.02
	4-CP	3.2	0.011	0.982	0.09	0.12	0.559	0.997	0.04
	2,4-DCP	6.9	0.012	0.977	0.22	0.24	0.588	0.998	0.07
Pine (4.75 mm)	2-CP	3.2	0.009	0.979	0.10	0.11	0.568	0.998	0.03
	4-CP	3.2	0.011	0.984	0.09	0.12	0.566	0.998	0.03
	2,4-DCP	6.8	0.013	0.980	0.21	0.26	0.575	0.999	0.05
HW (1.18 mm)	2-CP	3.9	0.011	0.984	0.10	0.16	0.544	0.997	0.04
	4-CP	4.0	0.013	0.973	0.13	0.19	0.527	0.998	0.03
	2,4-DCP	7.1	0.018	0.987	0.16	0.41	0.523	0.996	0.11

Equilibrium sorption experiments were carried out to determine the sorption capacity of pine and HW. The data were then fitted with Langmuir and Freundlich isotherms and evaluated for goodness of fit (Table 2). For both sorbents, the Freundlich model had a marginally better fit (higher r^2 and lower SSE) compared with the Langmuir model.

The isotherms were nonlinear with n (0.52–0.59) outside a linear range ($0.95 < n < 1.05$) [17] which indicates that at higher solute concentrations it becomes more difficult to sorb CPs onto wood.

The sorption capacities of the sorbents were evaluated based on the Freundlich K and Langmuir Q values with a greater K and Q value indicating enhanced sorption. The magnitude of sorption for the various CPs at equilibrium was 2,4-DCP > 4-CP > 2-CP across both sorbents, although there was relatively little contrast in the K value between the mono- and dichlorophenols. This sorption preference is consistent with other studies [18,19] that have found the adsorption capacity of phenolic compounds increases with an increase in solute hydrophobicity (e.g. K_{ow}). In biopolymers, it is the hydrophobicity of structurally similar organic compounds that is the main factor determining their ability to occupy sorption sites.[20]

Effect of particle size on sorption

The effect of particle size on CP sorption was evaluated for pine using four particle sizes (0.6–4.75 mm) and on HW (1.18 mm) (Figure 1).

Fitted sorption isotherm parameters for a particular compound are similar at equilibrium for all particle sizes (Table 2). This relatively uniform uptake trend is consistent with the similar surface area data for different particle sizes (Table 1). The poor correlation between particle size and surface area is due to the elongated nature of the particles.

The particle width rather than particle length was found to be more closely aligned with the sieve mesh opening. This is consistent with the elongate particles passing through the mesh vertically and has been reported from horizontally screened samples.[21]

Sorption mechanism

An intra-particle diffusion model was applied to kinetic data to give an insight into which mechanisms control solute uptake at various times. Numerous studies on sorption show that initial curved portions of the intra-particle diffusion plot suggest film diffusion processes while linear portions suggest intra-particle diffusion.[22] A third stage may occur where intra-particle diffusion decreases due to a low-solute concentration and the sorption rate plateaus. Weber and Morris [23] reported if a linear relation is obtained and that line passes through the origin, then intra-particle diffusion is the rate-controlling step. However, the rate-controlling mechanism may change during the sorption process.

The intra-particle diffusion plots (Figure 2) all have the same pattern. Both the pine and HW have relatively rapid initial uptake controlled by film diffusion followed by a curvi-linear stage which gradually plateaus. The shape and relative proportionality of these diffusion curves are similar showing that contaminant access into the particles is relatively similar and relatively independent of sorbent size.

Desorption

Equilibrium-based sorption–desorption experiments were conducted to determine the extent of irreversible sorption for 2-CP, 4-CP and 2,4-DCP from pine and HW. A plot of equilibrium solid-phase loading of sorption–desorption (q_e) against equilibrium liquid-phase CP concentration (C_e) of sorption–desorption is shown in Figures 3 and 4.

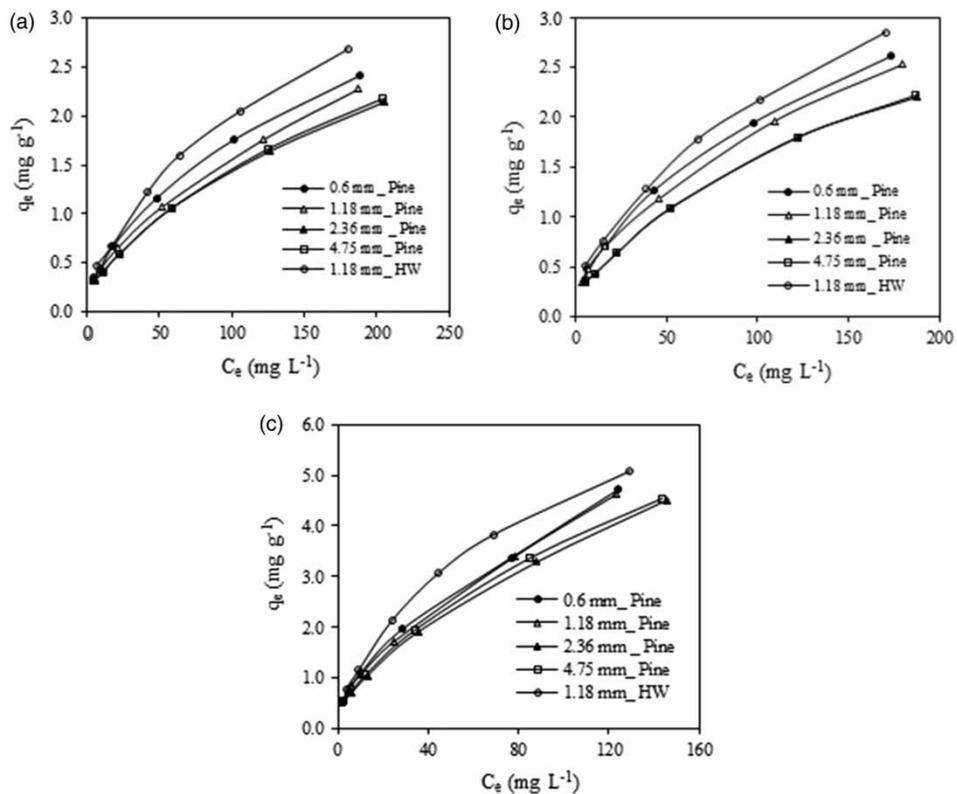


Figure 1. Equilibrium uptake (q_e) onto pine and HW for (a) 2-CP, (b) 4-CP and (c) 2,4-DCP at different particle size.

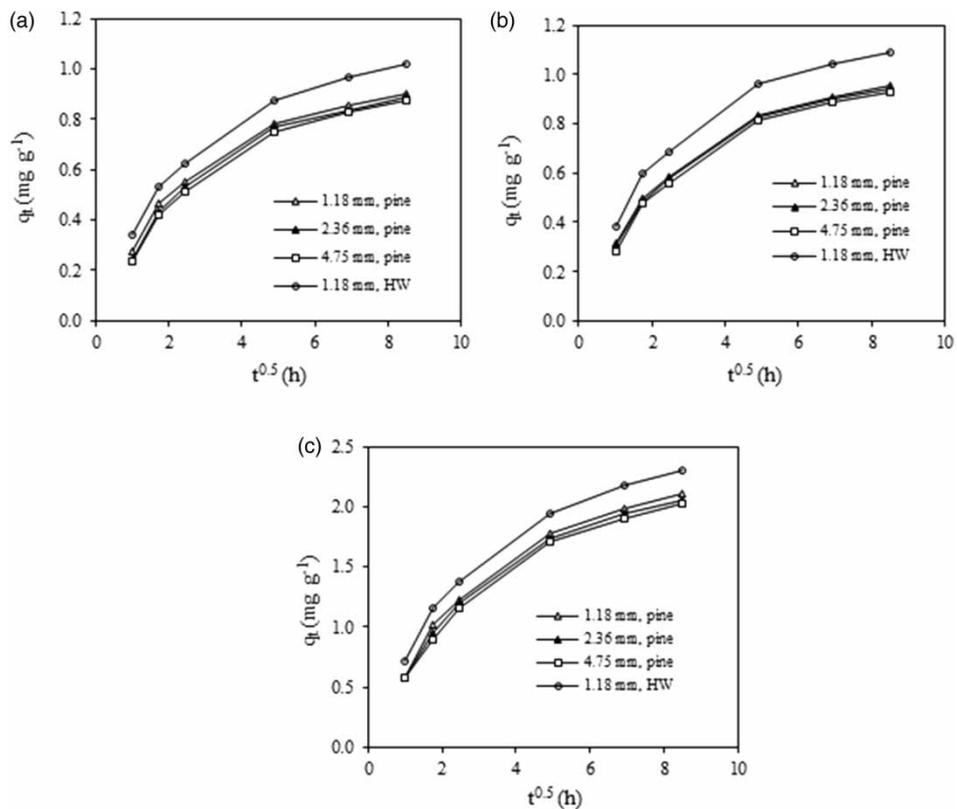


Figure 2. Uptake (q_t) onto pine and HW for (a) 2-CP, (b) 4-CP and (c) 2,4-DCP at various times ($t^{0.5}$).

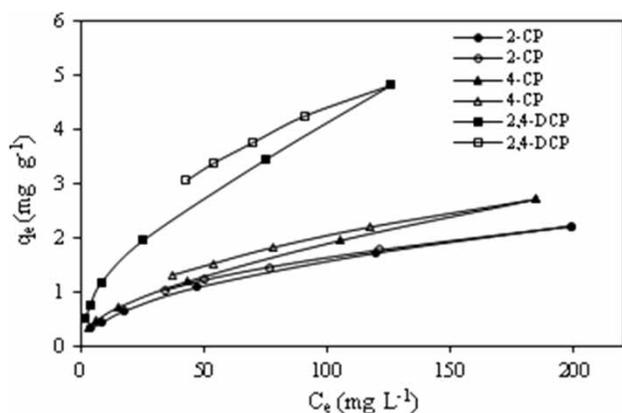


Figure 3. Sorption–desorption isotherms of CP by pine, 1.18 mm, solid symbols corresponds to sorption; open symbols corresponds to desorption.

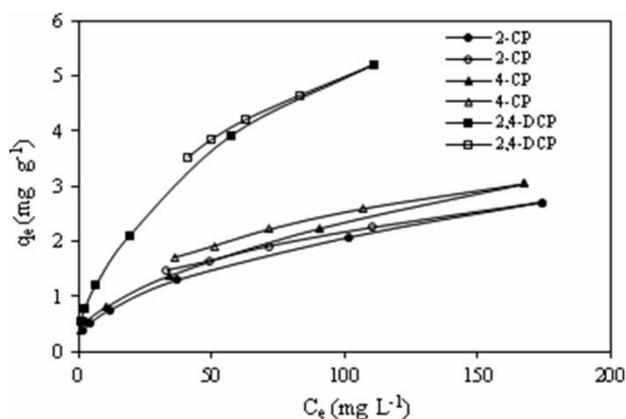


Figure 4. Sorption–desorption isotherms of CP by HW, 1.18 mm, solid symbols corresponds to sorption; open symbols corresponds to desorption.

The amount of equilibrium solid-phase loading after desorption was calculated by Equation (3). In case of fully reversible sorption both isotherms should coincide. In this study, all sorption isotherms lie below the respective desorption isotherm indicating the irreversible sorption (Figures 3 and 4). Experimental data were well fitted to the Freundlich equations for sorption and desorption behaviour. The sorption–desorption parameters for the Freundlich constants are given in Table 3.

For both pine and HW, the sorption capacity during desorption (K_d) was (1.46–1.8 times) greater than during

sorption (K). This indicates that there is a greater sorbed mass for a given solute concentration during desorption compared with that during a sorption cycle. Also, the desorption K_d values were consistently higher than those for sorption. This is indicative of sorption–desorption hystereses as a result of partially irreversible sorption of CPs onto a pine and HW. Adsorption–desorption behaviours can also be described in terms of hysteresis coefficients (H and λ). The results from the Table 3 showed that $H < 100$ and $\lambda > 0$, which indicate positive hysteresis in the pine and HW. The lowest hysteresis was observed for 2,4-DCP which has a high sorption capacity and reflects its strong hydrophobicity. These data indicate that the degree of irreversibility increases from 2-CP ~ 4-CP to 2,4-DCP.

Rodríguez-Cruz et al. [8] studied sorption–desorption isotherms of pesticides (^{14}C -Linuron) by untreated pine. They found that hysteresis coefficient for pesticides (^{14}C -Linuron) was 68. This is comparable with this study. The hysteresis coefficient in pine is lower for Linuron due to more hydrophobic ($\text{Log } K_{ow} = 3.0$) compared with pine for 2-CP and 4-CP which are less hydrophobic ($\text{Log } K_{ow} = 2.12\text{--}2.44$) and closer to pine for 2,4-DCP (almost similar hydrophobic, $\text{Log } K_{ow} = 2.75\text{--}3.3$). The hydroxyl group in phenolic is a strongly activating ortho- and para-directing substituent in electrophilic aromatic substitution reaction.[24] Therefore, electron density of a phenolic ring is strongly influenced by the nature of a substituent. Chlorine atoms act as an electron-withdrawing group in the phenolic ring. Thus, chlorine-substituted phenolic ring acts as acceptors in donor–acceptor complexes. Hence, more chlorine atom in the phenolic ring formed stronger complexes with a given donor. This might be the another plausible reason for higher irreversibility of 2,4-DCP.

Conclusions

Both pine and HW have potential as low-cost, low-efficiency sorbents for the removal of CPs from aqueous solution. Equilibrium capacity of pine did not significantly change with changing particle size. Greater pre-processing of these sorbents through size reduction may not be justified in terms of improved sorption capacity. The calculated hysteresis coefficient indicated that there is a hysteresis in pine and HW. Lower desorption by the more sorptive compound indicates that low hysteresis can be attributed to more

Table 3. Freundlich sorption (K and n) and desorption (K_d and n_d) parameters and hysteresis coefficients (H and λ) of CPs by pine and HW, 1.18 mm.

Sorbent	Sorbate	K	n	r^2	SSE	K_d	n_d	r^2	SSE	H	λ
Pine	2-CP	0.15	0.508	1.000	0.01	0.22	0.434	1.000	0.01	85	5.16
	4-CP	0.16	0.537	0.999	0.03	0.25	0.459	0.999	0.01	85	5.35
	2,4-DCP	0.34	0.543	0.998	0.07	0.62	0.424	0.999	0.02	78	8.36
HW	2-CP	0.25	0.458	0.998	0.04	0.38	0.378	0.998	0.02	83	5.81
	4-CP	0.29	0.454	0.994	0.08	0.43	0.381	0.999	0.01	84	5.29
	2,4-DCP	0.50	0.501	0.997	0.09	0.85	0.385	0.999	0.02	77	8.38

irreversibility due to strong hydrophobic interaction of 2,4-DCP with wood surfaces. This knowledge further improves their cost-effectiveness for field-scale applications.

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