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Effect of MW and pH on poly(ethylene glycol) adsorption onto carbon

Loris Pietrelli

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Abstract Poly(ethylene glycol) (PEG) is a water-soluble polymer commonly found in industrial and domestic wastewaters. In this study the adsorption onto granular activated carbon (GAC) of PEG, of different molecular weights, from aqueous solutions was examined to evaluate its applicability to wastewater treatment. Batch kinetic models have been tested to predict the rate constant of adsorption. The amount of PEG adsorbed on activated carbon depends mainly on the pH, the MW and on the solution characteristics. The adsorption at fixed temperature decrease by MW (PEG-8000 < PEG-3350 < PEG-1450) a polymer chain conformation modification can explain these effect. The large values of adsorption capacity (>350 mg/g) at low and high pH values show a great potential for GAC. The adsorption process can be described well with the Langmuir and the pseudo first order equation. The effective intraparticle diffusion coefficients of PEG molecules in the GAC adsorbent varying according to the MW values in the range 8.45×10^{-3}–9.71×10^{-7}.

Keywords Poly(ethylene glycol) · Adsorption · Granular activated carbon · Water treatment

1 Introduction

Poly(ethylene glycol) (PEG) is a highly water-soluble, waxy solid that is used extensively in the cosmetic, pharmaceutical, food, textile, polishing and toiletry industries. Therefore PEGs are commonly found in industrial and domestic wastewaters, as non-ionic surfactants coupled with hydrophobic molecules or as catabolic products of these. Several studies (Dwyer and Tiedje 1986; Watson and Jones 1976) have been performed on the biodegradability of PEG of various molecular weights and they have shown that biodegradation substantially decreases with increasing MW, thus making high MW polymers practically non-biodegradable.

There are two principal processes for the physico-chemical removal of PEG from wastewater: destructive processes such as ozonation (Andreozzi et al. 1996; Li et al. 2000), ozonation/UV (Chang et al. 2001), or H_2O_2/UV oxidation (Oliveros et al. 1997), and recovery processes such as filtration (Byhlin and Jonson 2002), adsorption onto activated carbon (Chang et al. 2004; Arbuckle and Osman 2000), montmorillonite clays (Zhao et al. 1989), suspended alumina (Zhang 2003), zinc oxide (Liufu et al. 2004) or zeolite (Chang et al. 2003). Chang et al. (2000) examined the adsorption equilibrium of PEG having large molecular weight from copper electroplating solutions on activated carbon at 288–313 K, and investigated the feasibility of removing the organic additive from the bath.

The adsorption behaviour of polymers (and surfactants) on solids strongly depends, generally, on the polymer solution characteristics (i.e. as PEG molecular weight increases, so the polarity weakens and therefore its affinity to water molecules decreases). According to the adsorption studies and considering its large surface area and non-polar surface, there is no ideal universal activated carbon for every application. Hence, it is important to match the properties of the activated carbon with the performance requirements of the process. Following a previous study where temperature, concentration effect and the breakthrough curve were considered (Gajdos et al. 2007), the

L. Pietrelli (✉)
ENEA-UTTAMB, CR Casaccia, Via Anguillarese 301,
00060 Rome, Italy
e-mail: loris.pietrelli@enea.it

influence of MW and pH on the adsorption of PEG onto activated carbon was studied.

2 Materials and methods

The commercial granular activated carbon (GAC) 207-EA for water treatment was supplied by B.M.D. S.r.l., (Bagni di Tivoli, Italy). Before using and characterizing, the adsorbent material was washed with distilled water to remove the crushed carbon. It was dried overnight in a vacuum oven at 80 °C, and then stored in a desiccator. Surface area measurements were made using the BET technique. The main physical properties of the GAC are given in Table 1.

Polyethylene glycol standards for adsorption experiments were purchased from Sigma-Aldrich (USA) and in particular experiments were performed with molecular weight of 1450, 3350 and 8000 respectively (Polydispersity Index, $M_w/M_n \approx 1.03$). Standard PEG solutions ranging from 0.2 to 5.0 g/l for each MW were prepared.

Adsorption of different MW PEG onto GAC was studied by doing batch experiments: fixed amounts of GAC (500–700 mg) were placed with a known volume (100 ml) of PEG solution into a beaker using a temperature-controlled water bath ($T = 20$ °C). After that it was brought into equilibrium by mechanical shaking until the equilibrium conditions were established after 3 h. PEG-8000, unlike the other MWs, shows a difference of 3–4 % in the adsorption capacity compared to the data obtained after 24 (Gajdos et al. 2007).

The supernatant was filtered using a 0.45 μm Millipore membrane then the PEG concentration was determined using a VARIAN TECHTRON DMS 200 spectrophotometer at 192.4 nm wavelength (the spectral properties of PEG are independent of pH and show a high absorbance within the 190–230 nm range). The concentration of residual PEG was also determined using a total carbon analyzer (TOC) (DC–190 Rosemount Analytical Inc., USA) to confirm the spectrophotometer analysis. The correspondence between the PEG concentration and the TOC was: 1 mg PEG = 0.57 mg TOC.

The amount of the adsorbed PEG, qe (mg/g), was determined as:

$$qe = (C_0 - C_e)V/W$$

where C_0 and C_e (mg/l) are respectively the initial and equilibrium liquid-phase concentrations of the PEG solution; V is the volume (ml) of the PEG solution and W is the mass (g) of the dry GAC sample. The pH effect on PEG removal was studied by adjusting the pH of the test solution using 0.1 N HCl or 0.1 N NaOH solutions.

3 Results and discussion

3.1 Kinetic studies

There is a strong affinity of PEG and relative fast adsorption rate (Gajdos et al. 2007). The adsorption rate decreases with time probably due to a decrease in the concentration driving force and the difficulty of molecular diffusion. The molecular weight seems to play an important role in controlling PEG adsorption. Data regarding low MWs and higher concentration samples clearly show an increase in adsorption capacity, especially for the lowest MW. This is probably due to poor polymer hydration rate. The more the concentration increases, the more the hydration number decreases for all the chain lengths. At high polymer concentration it reaches nearly zero level (Dormidontova 2004). In general, hydration (per repeat unit) increases for the shorter PEG chain lengths in the whole concentration range. The relative contribution of end group hydration is larger compared to longer chains and consequently the hydration number is greater.

The adsorption process on a porous adsorbent generally involves several transport stages such as external and internal diffusion, and the adsorbate behaviour in the solution plays an important role in the adsorption mechanism. The conformation of any polymer in solution is affected by a

Table 1 Main characteristics of the 207 EA granular activated carbon

Surface area (BET N_2) (m^2/g)	990	Iodine number (mg/g)	1,250
Humidity, 60 °C, 2 h (wt%)	2.2	Molasses index	220
Water soluble ash (wt%)	<1	Methylen blue index, ASTM D-2330 (mg/g)	114–182
Hardness (%)	93–96	pH	9
Bulk density (g/cm^3)	0.52	Micropore volume (cc/g)	0.472
Free flowing density (g/cm^3)	0.45	Mesopore volume (cc/g)	0.120
Average pore size (nm)	2.2	Macropore volume (cc/g)	0.255
Particle size (mesh)	12 × 40	Total pore volume (cc/g)	0.847
Carbon tetrachloride index (wt%)	110		

number of factors such as polymer architecture and solvent affinity; an uncharged linear polymer chain is usually found in solution in a random conformation loosely approximating a three dimensional random self-avoiding walk (according to the Flory-like theory as reported by De Gennes 1976). Although many theoretical model equations have been proposed to describe the adsorption kinetics based on mass balance, pore diffusion rate and boundary conditions, the large amount of oxygen on the PEG chain and the polymer MWs, would probably make any kinetic or mass transfer representation global. In this study the simplest kinetic analysis of adsorption was performed.

The pseudo-first-order equation described by Lagergren (1898) can be rearranged to obtain a linear form:

$$\log(q_e - q_t) = \log\ q_e - (k_1/2.303)t$$

A pseudo-second-order equation according to Ho and Chiang (2001) may be expressed in the form:

$$t/q_t = (1/k_2 q_e^2) + (1/q_e)t$$

As the two equations above cannot give definite mechanisms, the simplified initial rate of intraparticle diffusion model according to Weber and Morris (1963) was tested:

$$q_t = k_i t^{1/2}$$

where q_e (mg/g) represents the amount of adsorption at equilibrium, q_t (mg/g) represents the amount of adsorption at time t, k_1 represents the rate constant of the pseudo first order kinetic model, k_2 rate constant of the pseudo second order kinetic model and k_i is an intraparticle diffusion rate constant.

The fitting validity of these models can be checked by the slopes and intercepts from each linear plot of $\log(q_e - q_t)$ vs t, t/q_t vs t and q_t vs t 1/2 respectively.

Table 2 shows the results for each MW at different PEG concentrations when comparing the correlation coefficients (R^2), the calculated and experimental q_e values.

The correlation coefficient R^2 for the pseudo first order and intraparticle diffusion adsorption models have extremely high values and their calculated equilibrium adsorption capacity ($q_{e,theor}$) fit very well with the experimental (qe_{exsp}) values at the fixed pH value. This is not surprising since it is known that even diffusion limited processes can be fitted with the first-order model. This also suggests that the overall rate of PEG adsorption process does not appear to be controlled by a chemisorption process (also indicated by the relatively low values of k_2).

3.2 Diffusion coefficients

In many cases the resistance to internal diffusion can be extremely significant. However, the local adsorption rate is assumed to be relatively fast and the resistance to external diffusion negligible if compared to intraparticle diffusion. Taking into account that the effect of the agitation speed seems to be negligible for the PEG solution/zeolite

Table 2 Values of adsorption rate constants varying MW and PEG concentration, T = 20 °C, pH 5

		Pseudo-first order			Pseudo-second order			Intrap. diffusion	
	Qe_{exsp}	qe_{theor}	k_1	R^2	qe_{theor}	k_2	R^2	qe_{theor}	R^2
MW1450 (g/l)									
0.5	83.29	98.70	0.01152	0.961	558.05	1×10^{-5}	0.703	80.09	0.964
1	140.36	163.53	0.01198	0.973	256.41	2×10^{-5}	0.652	136.82	0.975
2	156.50	181.93	0.01935	0.935	204.08	7×10^{-5}	0.996	172.26	0.982
MW3350 (g/l)									
0.2	22.44	26.27	0.00714	0.881	50.76	4×10^{-5}	0.857	20.89	0.963
0.5	73.08	70.7	0.01013	0.962	222.09	6×10^{-6}	0.179	63.72	0.902
1	92.04	91.83	0.01359	0.956	119.05	9×10^{-5}	0.963	95.79	0.912
1.5	106.9	121.48	0.01059	0.979	222.22	1×10^{-5}	0.675	113.9	0.941
2	104.76	112.12	0.01059	0.980	136.99	7×10^{-5}	0.999	112.2	0.981
3	113.94	107.03	0.01105	0.988	136.99	1×10^{-4}	0.997	312.89	0.912
MW8000 (g/l)									
0.2	35.16	23.57	0.01566	0.940	37.31	1×10^{-3}	0.999	40.38	0.955
0.5	74.72	73.15	0.01451	0.971	90.09	2×10^{-4}	0.996	82.43	0.955
1	85.52	98.27	0.01082	0.979	332.58	4×10^{-6}	0.243	87.34	0.933
1.5	75.86	96.69	0.01198	0.961	285.71	5×10^{-6}	0.309	77.32	0.945
2	82.94	42.79	0.01842	0.986	151.52	2×10^{-5}	0.943	77.64	0.985
3	102.68	85.33	0.00898	0.969	121.95	1×10^{-4}	0.990	260.93	0.913

Table 3 Diffusion coefficients and adsorption half time

PEG MW	D_b (cm^2/s)	D_e (cm^2/s)	$t_{1/2}$ (min)
1450	8.45×10^{-3}	0.43×10^{-3}	51.0
3350	1.60×10^{-6}	0.82×10^{-7}	80.1
8000	9.71×10^{-7}	0.22×10^{-8}	94.1

adsorption system (Chang et al. 2003), we can deduct that the adsorption is controlled by transport within the pore network and by the strong hydrodynamic interactions caused by hydrogen bonding of PEG chains in water.

For the PEG diffusion the following equation can be used (Smith 1986):

$$De = [D_b \varepsilon/\tau] F_\lambda$$

where D_b is the bulk diffusion coefficient or diffusivity in free solution, ε is the porosity, τ is the tortuosity factor (τ = 3.5 for similar GAC, Leyva-Ramos and Geankoplis 1994) of the porous particle ranging from 2 to 7 (Satterfield 1991), and F_λ is a function of the ratio of critical molecular—and pore size. This accounts for the hindrance/exclusion and hydrodynamic drag effect. A rough estimate can be given of the critical molecular size (minimum cross section diameter) of PEG (Chang et al. 2003). Therefore considering the average pore diameter and the Renkin (1954) expression for the restrictive diffusion effect, F_λ can be determined.

The bulk diffusivity of PEG was estimated using the following equation (Shao and Baltus 2000):

$$D_b = 1.465 \times 10^{-4} \times \ MW^{-0.557}$$

The D_b values are reported in Table 3 and the value obtained for PEG-8000 is in good agreement with those reported by Shao and Baltus (2000) for the PEG-10000 (7.4×10^{-7} cm^2/s). The highest D_b value obtained for the PEG-1450 may also justify adsorption half time (of the PEG), $t_{1/2}$, that is the time required for the adsorbent to take up half of the max adsorption capacity, q_e as obtained from the kinetic equations, that is the shortest time to reach the equilibrium at the same polymer concentration (Table 3).

The external mass transfer is usually negligible when pore diffusion is the controlling intraparticle diffusion mechanism. The comparison of the Diffusion coefficients shows that the adsorption of the highest MW seems to be affected by intraparticle diffusion.

The effect of temperature (Gajdos et al. 2007) and pH clearly shows that there is an aggregation of polymer-water and polymer-water-polymer. However the values of the effective diffusion obtained need further work in order to elucidate bulk, surface and pore diffusion such as the cross section data were obtained considering the unperturbed dimension of a PEG chain (e.g. using a hydrophobic organic solvent such as benzene).

3.3 Adsorption isotherm

The adsorption process generally proceeds through varied mechanisms such as external mass transfer of solute onto sorbent followed by intraparticle diffusion, making it impossible to determinate the rate-controlling step. Moreover, conformation of the adsorbate on an adsorbing surface can complicate the mechanism interpretation. It can be likened to *trains*, whose segments are in contact with the adsorbent (*loops*), but not in contact with the surface and whose function is to connect two trains and *tails*—the unadsorbed *chain end* (Fleer et al. 1993). Therefore empirical design procedures based on adsorption equilibrium conditions are the most common method to predict adsorber size and performance. The most common adsorption models were compared: the Langmuir (1) and Freundlich (2) equations.

The Langmuir isotherm is as follows:

$$q_e = Q^0 bC_e/(1 + bC_e) \\ \text{or, linearized } 1/qe = \left(1/Q^0 K_L\right)(1/C_e) + 1/Q^0 \tag{1}$$

where q_e (mg/g) is the amount of element adsorbed per unit weight of adsorbent, Q^0 (mg/g) is the solid phase concentration corresponding to complete coverage of available sites (limiting adsorption capacity),

C_e (mg/l) is the residual liquid phase concentration at equilibrium, K_L is the adsorption coefficient.

The linearized Freundlich equation in logarithmic form:

$$\log q_e = \log K_f + 1/n \log C_e, \tag{2}$$

where K_f and 1/n are characteristic constants, which are respectively determined from the intercept and slope of the Freundlich equation on the logarithmic plot, and which respectively represent the sorption capacity and adsorption intensity.

According to the Freundlich model, the slope 1/n, ranging between 0 and 1, is a measure of the adsorption intensity or surface energy heterogeneity, becoming more heterogeneous as its value gets closer to zero. In Table 4 the empirical constants from the linearized Langmuir and Freundlich equations are reported for pH values of 3, 5 and 8. When comparing the correlation coefficients, the results obtained from the Langmuir model give a satisfactory correlation between the model predictions and the experimental data for each pH values therefore the monolayer coverage of solute particles onto micro-porous GAC can be assumed.

Table 4 Isotherm model parameters for adsorption process of PEG onto activated carbon at different pH values

	Langmuir				Freundlich		
Curve	Q_0 (mg/g)	K_L	R^2	R_L	K_f (mg/g)	1/n	R^2
PEG-1450, pH 3	388.41	8.58	0.9964	0.028	353.32	0.3540	0.8996
PEG-3350, pH 3	301.02	6.64	0.9462	0.036	320.51	0.1808	0.6653
PEG-8000, pH 3	239.38	20.89	0.9683	0.005	245.30	0.2869	0.9024
PEG-1450, pH 5	188.68	5.88	0.9836	0.055	144.97	0.3396	0.7328
PEG-3350, pH 5	126.58	3.16	0.9744	0.096	79.82	0.5905	0.7801
PEG-8000, pH 5	81.30	24.60	0.9849	0.013	81.99	0.2104	0.7826
PEG-1450, pH 8	250.71	6.67	0.9941	0.025	267.43	0.3231	0.8843
PEG-3350, pH 8	230.83	5.93	0.9804	0.032	241.53	0.2143	0.8901
PEG-8000, pH 8	200.64	19.54	0.9798	0.010	198.73	0.2340	0.8753

The essential characteristics of the Langmuir equation can be expressed in terms of a dimensionless separation factor R_L which is defined by McKay (1982) as: $R_L = 1/[1 + (K_L C_o)]$ where C_o is the highest initial polymer concentration (mg/ml). The value of R_L indicates the shape of the isotherm to be either unfavourable ($R_L > 1$), linear ($R_L = 1$), favourable ($0 < R_L < 1$) or irreversible ($R_L = 0$). In Table 4 the calculated separation factor values are given for each PEG MW. In fact, the low K_L values seem to confirm the high affinity between GAC and PEG.

Considering the Langmuir assumption of the polymer monolayer structure, the dependence of the maximum value of PEG adsorption from aqueous solutions on the GAC surface on the polymer mass can be described by a linear equation (Eltekov et al. 2004): $\log qe_{max} = a + b \log MW$. In the case of weak dependence of qe on MW ($b \approx 0$), the thickness of the adsorption monolayer of the polymer does not change when its molecular mass increases.

The analysis of dependences according to the *b* values calculated for each MW at different PEG concentrations are between 0.06 and 0.37, indicating the dependence of maximum adsorption on polymer molecular mass. It is likely that the adsorbed polymer monolayers of identical thickness are composed of unfolded PEG macromolecules (probably as *train* conformation), which are distributed on the GAC surface. In addition, the presence of microporosity limits the surface accessibility to large conformations of polymer chains (aggregate or solvated molecules). This can also be explained by the strong effect of pH and temperature on the polymer conformation in aqueous solution.

3.4 Effect of the pH on the PEG adsorption

Adsorption of PEG with different molecular weights within a pH range of 2–11 was studied and the results are given in Fig. 1.

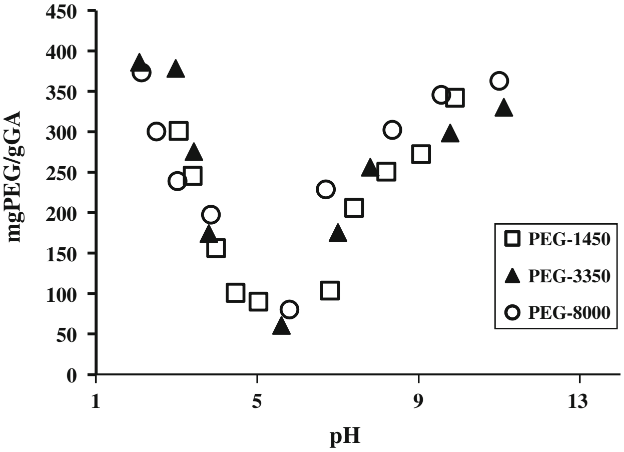

Fig. 1 pH effect on the PEG adsorption

The adsorption mechanism is mainly affected by the hydrophilicity of the carbon surface, and for non-ionic polymers, hydrogen bonding is the primary adsorption mechanism (Chang et al. 2000).

Active sites of PEG correspond to oxydril groups (–OH) situated at the chain ends and oxygen atoms from ether groups –CH2–O–CH2–, which are capable of specific interactions with adsorbents. Repulsive electrostatic interactions appear to have some influence on the adsorption of PEG. As a consequence, a decrease in the negative charge, or an increase in the positive charge on the activated carbon would decrease any surface-PEG repulsive interaction. According to the results obtained, the adsorption of PEG is strongly dependent on pH and the best pH for the adsorption process with activated carbon is in the range of 2–3.

This behaviour is a likely consequence of the nature of the polymer chain with its hydrophobic methylene groups interspersed with ether groups which can complement the hydrogen bonded structure of water. Therefore, the hydration/dehydration behaviour of these groups can promote the observed behaviour. Moreover, the low solvation rate of PEG promotes pore, film and bulk diffusion and presumably, the less bulky form occurs at these pH values.

Likewise, the stability constant increase with the increment of the PEG MW observed for the Na^+-PEG complex (Chen et al. 2005) may explain the sorption capacity observed for the H^+-PEG-8000 system.

An interesting behaviour of the PEG aqueous solutions to consider is the observed pH increase with the polymer concentration and the MW increasing. The basicity of the R-O-R oxygen (much more than the oxydril group at high MW) may justify this behaviour. The coincidence of the Zero Point Charge at pH 8–9 for both GAC and PEG solutions may justify the increment of adsorption at $pH > 7$ (no repulsion forces occur).

4 Conclusions

Polyethylene glycols at different molecular weight can be effectively removed from the water solution by adsorption onto activated carbon. The adsorption process in particular fits the Langmuir isotherm model as reflected by correlation coefficients which ranged from 0.97 to 0.98. The PEG adsorption capacity increase with initial concentration (C_i), with equilibrium concentration (C_{eq}) and decrease with PEG MW. Adsorption of polyethylene glycol is strongly dependent on the pH, the best results of adsorption have been achieved in the range of pH 2–3. The pH effect probably can be correlated to the polymer chain conformation. The kinetic studies shown that the intraparticle diffusion and Pseudo-first order models better describe the adsorption rate. According to the literature, the adsorption capacity of GAC is larger than those obtained from other adsorbents such as silica, zeolite and alumina. As a result it is feasible to use GAC to remove PEGs from aqueous solutions even at high polymer concentrations, such as the electroplating wastewater at low pH.

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