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ADSORPTION CHARACTERISTICS OF DIRECT BLUE 78 ONTO POLYETHYLENE GLYCOL GRAFTED POLYSTYRENE RESIN

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ABSTRACT

A comparison of adsorption characteristics of polyethylene glycol grafted polystyrene (PS-*g*-PEG) resin and activated carbon (F-400) on Direct Blue 78 was carried out. The PS-*g*-PEG showed significantly higher adsorption capacity and adsorbed more rapidly than F-400. Adsorption on PS-*g*-PEG was highly influenced by salt concentration, but was relatively unaffected by pH change. Dye desorption from PS-*g*-PEG was achieved relatively easily. The different adsorption characteristics of PS-*g*-PEG resin and F-400 are explained in terms of the physical structure and the chemical properties of each adsorbent.

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Key Words: Polyethylene glycol grafted polystyrene resin; Direct Blue 78; F-400; Adsorption; Desorption

INTRODUCTION

Nowadays, many chemists and biologists have shown interest in polyethylene glycol grafted polystyrene (PS-*g*-PEG) resin, in the context of building peptide libraries or small organic molecule libraries using solid-phase strategy for the development of therapeutic agents, because it has several advantages compared to conventional PS resin (1,2). The main advantages of PS-*g*-PEG resin are its high swelling characteristics in various organic solvents and in aqueous system, good compatibility with biological systems, and good solvation of hydrophobic and hydrophilic compounds. The high swelling properties of PS-*g*-PEG resin may induce a reagent to diffuse quickly into the resin with solvent, and this proceeds smoothly in a more homogeneous phase (3). Another application of PS-*g*-PEG resin is found in phase-transfer catalysis as previously reported in Refs. (4,5). Unlike the conventional soluble phase-transfer catalyst, PS-*g*-PEG resin can be easily removed from reaction mixtures, and is proven to be a very effective catalyst in bi-phasic reaction systems (6).

Activated carbon has been used as a popular adsorbent in waste or wastewater treatment systems. However, there are still several limitations associated with its use, such as its poor ability to adsorb hydrophilic compounds and its eventual disposal (7). Synthetic resins are used in a number of industries as alternatives, since they can be easily regenerated and the chemical pollutants can often be recovered (8). Previously, it was reported that PS-*g*-PEG resin adsorbed dye molecules effectively in acetonitrile (9). However, no further report that deals with the adsorption of dye in aqueous solvent is available. As PS-*g*-PEG resin is amphiphilic and swells very well in water (3), there is a good possibility that it could be used as an adsorbent for adsorbing a variety of synthetic organic chemicals. In this study, we investigated the possibilities of using PS-*g*-PEG resin as an adsorbent, and reported upon the influences of several important factors (pH, salt concentration, and mixing time) on the adsorption properties of PS-*g*-PEG resin. The desorption properties of PS-*g*-PEG resin were also examined.

EXPERIMENTAL

Preparation of 2-(1-Methyl)Hydroxyethyl Polystyrene Resin (10)

As a precursor of PS-*g*-PEG resin, 2-(1-methyl)hydroxyethyl PS resin (PS–PO) resin was prepared from PS resin by Friedel–Crafts alkylation with propylene oxide in the presence of $SnCl_4$ catalyst. After thorough washing (11), PS–divinylbenzene (1%) copolymer resin (Bio-Rad Co., USA, BioResin S-X1, 200–400 mesh, 100 g) was swollen in methylene chloride (1.5 L) in a 3-necked round-bottomed flask, and $SnCl_4$ (30.9 mL, 201.6 mmol) was added to the resin mixture in an ice bath with mechanical stirring (300 rpm). Propylene oxide (18.5 mL, 201.6 mmol) in 100 mL of methylene chloride was then added drop wise with stirring. After 12 hr of reaction, 100 mL of methanol was added to quench the remaining catalysts and the resins were washed with methanol (× 2), 1 *N* aqueous HCl/dioxane (× 2), water/dioxane (× 3), methylene chloride (× 2) methanol (× 2), and dried in vacuum.

Preparation of Poly(Ethylene Glycol)-Grafted Polystyrene Resin (10)

The PS–PO resin (100 g) and KOH (11.2 g, 199.6 mmol) were added in purified dioxane (150 mL) into a pressure reactor (Taiasu Co., Japan) and purged with nitrogen gas for 10 min at 100°C to remove moisture. Ethylene oxide (510 g, 11.6 mol) was then added to the reactor and the reaction mixture was maintained at 120°C with mechanical stirring. After 5 hr, the resins were filtered and washed with dioxane (× 2), 1 *N* aqueous HCl/dioxane (× 2), water/dioxane (× 3), methylene chloride (× 2) methanol (× 2), and dried in vacuum. The terminal hydroxy groups of the PS–PO resin were determined by Dimethoxytrityl chloride (DMT-Cl) titration (12).

Dye Adsorption Experiments

In addition to the PS-*g*-PEG resin, one commercially available activated carbon (F-400, Calgon Co., USA) was used as an adsorbent for comparison purposes. Direct Blue 78 (Solophenyl Blue 4GL; CI No. 34200; approximately 50% purity, molecular weight = 1059.95, supplied by Novartis [Ciba-Geigy Co., Switzerland)] was selected as a model dye. Since only approximate purity of this dye was available from the supplier, the adsorption capacity obtained from the dosage of the dye applied was presented. Therefore, careful consideration reflecting the purity of dye is required in interpreting the adsorption capacity obtained from this study. The chemical structure of Direct Blue 78 (DB78) is shown in Fig. 1.

The initial dye concentration was fixed at 250 mg/L. Anhydrous sodium sulfate (Na_2SO_4) was used as the salt (0, 10, 50 g/L), and pH 4 (0.05 *M* sodium acetate) and 7 (2.5×10^{-3} *M* potassium phosphate) sample solutions were made up according to the standard method (13). The pH 10.0 buffer was purchased from Duksan Chemical Co. (Korea).

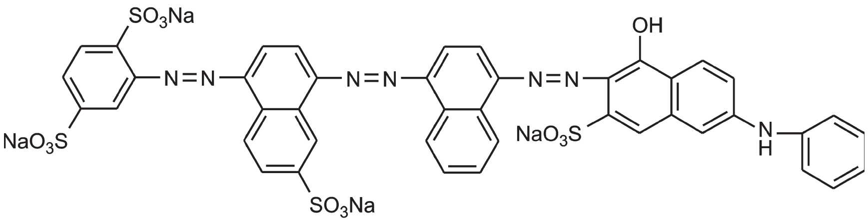


Figure 1. The chemical structure of Direct Blue 78.

The F-400 (activated carbon) was crushed to 80–100 mesh (150–180 μm) to achieve a size similar to that of the PS-*g*-PEG resin. Each adsorption isotherm of PS-*g*-PEG and F-400 was obtained as the concentration of the two adsorbents varied from 150 to 1250 mg/L with 200 mL of dye solution in a lab-made tumbler. After mixing for 10–12 hr, the samples were filtered using glass microfiber filters GF/C (Whatman Co., England), and their absorbances were measured at $\lambda_{\max}$ (604 nm at pH 7) using a UV spectrophotometer (HP 8452 A diode array spectrophotometer, Hewlett Packard Co., USA) with confirmation of no significant adsorption of Direct Blue 78 on the surface of glass microfiber filters GF/C. The change of pH under this study did not cause any significant change in absorbance and $\lambda_{\max}$. Absorbances were converted to concentrations using a calibration curve. The readings reported in the figures are all just single readings. However, several replicate experiments in representative conditions (i.e., salt 10 and 50 g/L at pH 7) were performed to check the reproducibility of these experimental works. All the replicate data were found within 10% difference (not shown in the text).

RESULTS

Properties of PS-*g*-PEG Resin

PS-*g*-PEG resin beads are grafted copolymers, which consist of a cross-linked (1%) PS matrix grafted with PEG. As PEG is an amphiphilic polymer, i.e., one with hydrophobic and hydrophilic properties, the grafted copolymer shows modified physico-chemical properties. The initial substitution level of the hydroxy group used was 1.27 mmol/g, which means that quantitatively one in eight of the phenyl rings was grafted with 2-(1-methyl) hydroxyethyl groups.

PS-*g*-PEG resins were prepared from PS–PO resin by anionic graft polymerization of ethylene oxides to the terminal hydroxy groups of PS–PO resin. After treating PS–PO resin with KOH as catalyst in a pressure reactor, ethylene oxides were added and polymerized to yield PS-*g*-PEG resins. Table 1

Table 1. Properties of Polyethylene Glycol Grafted Polystyrene Resin Beads

Initial Substitution Level (mmol/g of PS–PO)[a]	Final Substitution Level (mmol/g of PS-*g*-PEG)[a]	Added PEG (g/g of PS-PO)[b]	Average M_w of PEG[b]	Fraction of PEG (%)[b]
1.27	0.29	3.39	2670	77.2

[a] The values were determined by DMT-Cl titration.
[b] These values were calculated from the initial and the final substitution level.

shows the properties of PS-*g*-PEG resins prepared for this study. The surface area of the resins by the Brunauer–Emmett–Teller method cannot be obtained, since PS-*g*-PEG resin has no definite surface other than the net of PEG or PS chain.

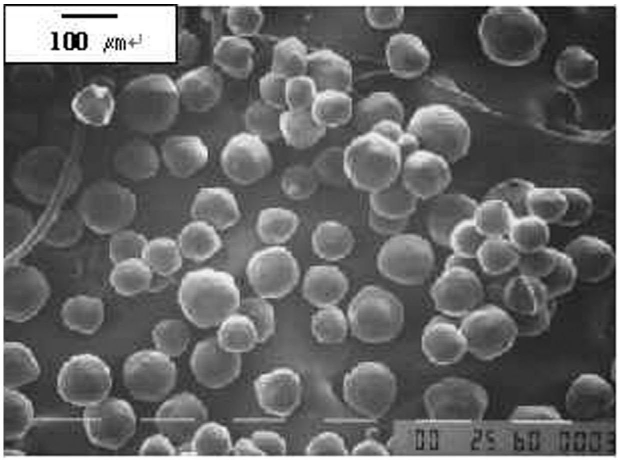
From the scanning electron microscope (SEM) pictures in Fig. 2, the size of the PS-*g*-PEG resin beads was 100–130 μm and they contained about 60 PEG units that comprise about 77.2% of PEG (w/w). Surface morphologies were changed from smooth to orange skinlike by the grafting procedure.

Preliminary Adsorption Comparison of PS-*g*-PEG and F-400

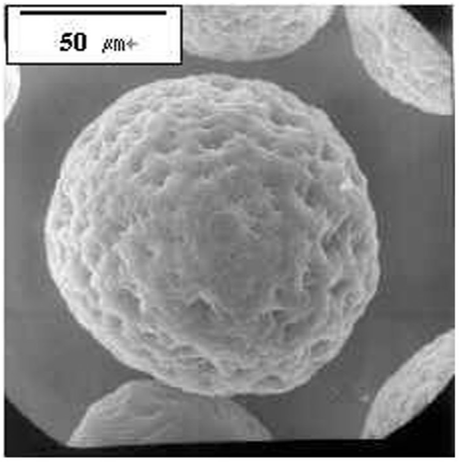
Isotherm testing was performed to find out the maximum adsorption capacity of the adsorbent, and one of the representative results is shown in Fig. 3. Schematic curves in the figure were included for better representation of experimental results. Direct Blue 78 solution and each adsorbent were mixed at pH 7 without a salt. The equilibrium concentration of Direct Blue 78 was measured after 10 hr. Figure 3 shows that PS-*g*-PEG resin showed a much better adsorption capacity than F-400. The PS-*g*-PEG adsorbed more than 10% by weight of Direct Blue 78 at an equilibrium concentration of 150 mg/L Direct Blue 78, and this amount was 1.8 times more than that absorbed by F-400.

Effect of Salt and pH

The effect of salt concentration on dye adsorption is especially important since dyes in wastewater effluents are frequently found in solutions with high salt concentrations. The effects of salt concentration and pH on the adsorption capacity of PS-*g*-PEG were investigated. The isotherm tests were performed at various salt concentrations (Fig. 4, pH 7) and at various pHs (Fig. 5, 10 g/L salt concentration). The results shown in Fig. 3 were included in Figs. 4 and 5 for



(a)



(b)

Figure 2. Scanning electron microscope of PS-*g*-PEG resin bead: (a) × 25; (b) × 500.

comparison. The different adsorption capacities of PS-*g*-PEG and F-400 are better shown in Fig. 4 at high salt concentration. Higher adsorptions by the two adsorbents for Direct Blue 78 are generally expected at higher salt concentrations due to "salt effect." However, the extent of increased adsorption, due to the presence of salt differs greatly for the two adsorbents. Figure 4 shows that the adsorption capacity of PS-*g*-PEG was highly affected by salt concentration when

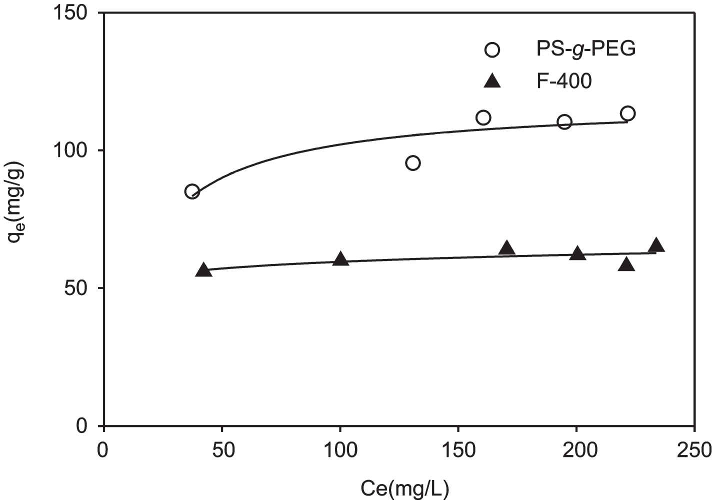


Figure 3. The result of isotherm test of PS-*g*-PEG and F-400 (pH 7, no salt).

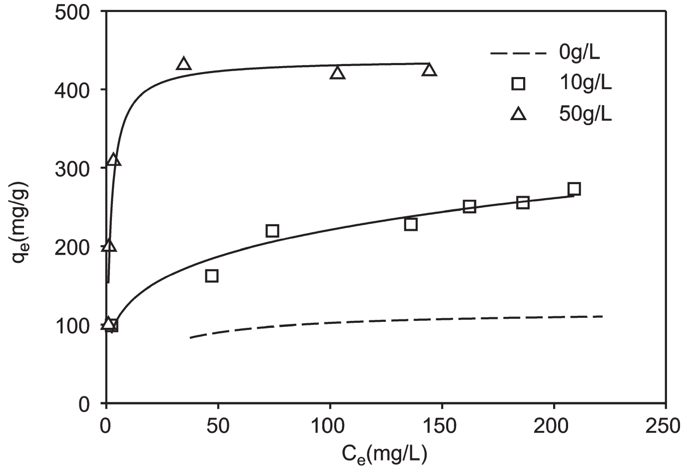
compared with F-400. The maximum adsorption capacities of PS-*g*-PEG for Direct Blue 78 at salt concentrations of 0, 10, and 50 g/L were 1.7, 2.7, and 3.7 times higher than that of F-400.

Adsorption Kinetics

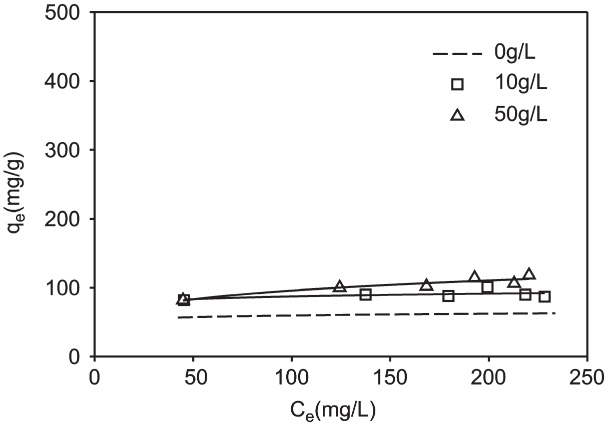
The dye adsorption kinetics of PS-*g*-PEG and F-400 were investigated over 30 min under the following conditions: 250 mg/L of initial dye concentration, 500 mg/L of adsorbent, and pH 7. Figure 6 shows that the concentrations of Direct Blue 78 in the presence of PS-*g*-PEG were reduced rapidly and almost reached an equilibrium concentration after 30 min, while dye levels were hardly reduced in the case of F-400. The effect of the presence of salt on the adsorption kinetics is also shown by Fig. 6. Only in the case of PS-*g*-PEG, was the presence of salt significant with respect to adsorption kinetics, which was consistent with the results shown in Fig. 4.

Comparison Between Polystyrene and PS-*g*-PEG Resins in Terms of Adsorption Capacity

Polystyrene is one of the components of PS-*g*-PEG, and constitutes about 20% of its weight. To examine the role of the constituent parts of PS-*g*-PEG resin with



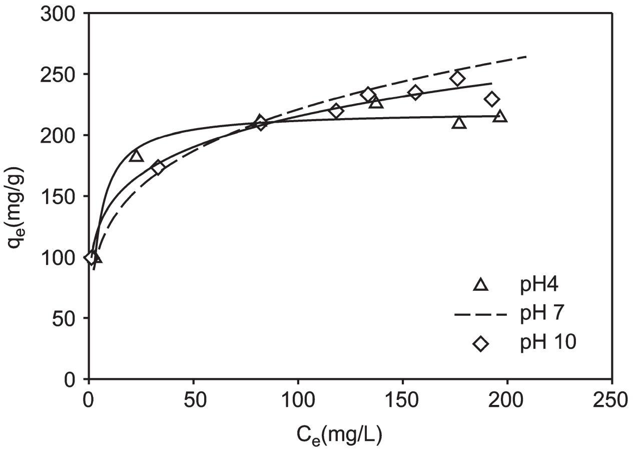
(a) PS-*g*-PEG



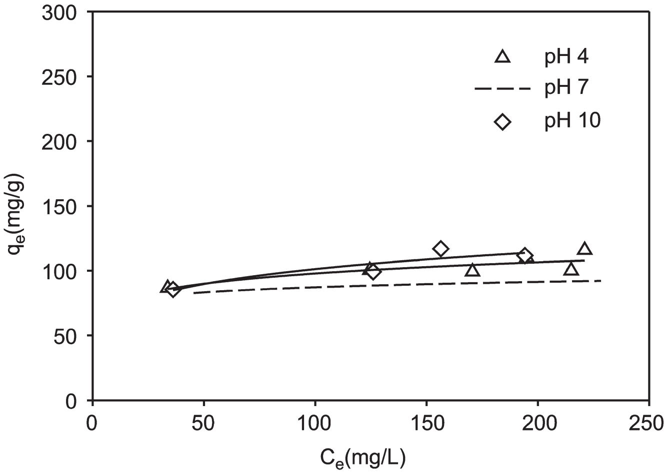
(b) F-400

Figure 4. The comparison of isotherm tests at various salt concentrations (pH 7).

respect to dye adsorption, comparison of adsorption capacities of PS-*g*-PEG and PS resin was carried out (Fig. 7). Bio-Resin S-X1 Resin (Bio-Rad) of particle size 40–80 μm and 1% crosslinked was used as PS resin. Isotherm tests were performed at 10 g/L salt concentration and pH 7. Figure 7 shows that the maximum adsorption capacity of the PS-*g*-PEG was about 3.7 times greater than that of the PS resin based on a weight for weight basis. This suggests that most of the Direct Blue 78 is adsorbed by the PEG component of the PS-*g*-PEG resin, and not by the PS component.



(a) PS-*g*-PEG



(b) F-400

Figure 5. The comparison of isotherm tests at various pH values (salt concentration of 10 g/L).

Dye Desorption from Adsorbent

Desorption tests with PS-*g*-PEG and F-400 were performed to examine the reversibility of the dye adsorption. Samples of PS-*g*-PEG and F-400 weighing 0.4 g, fully adsorbed with Direct Blue 78, were equilibrated in 200 mL of distilled

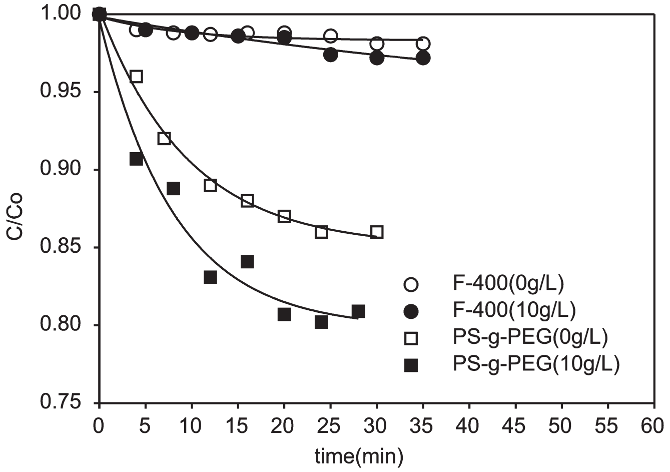


Figure 6. The dye adsorption kinetics.

water for 1 hr. The dye concentration adsorbed on the PS-*g*-PEG was 84 mg/g, and that on F-400 was 60 mg/g. The amount of dye eluted from each adsorbent was compared. The dye concentration desorbed from the PS-*g*-PEG was 45 mg/L, which corresponded to 73% of the total amount of dye adsorbed, and that desorbed from F-400 was 0.43 mg/L, which corresponded to 1% of the total dye

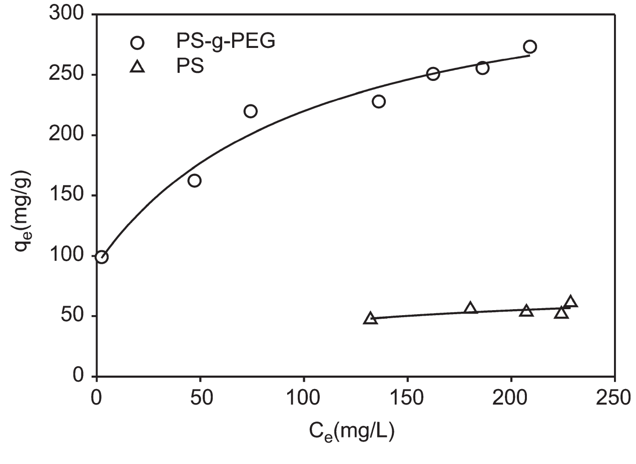


Figure 7. The role of PS or PEG component of PS-*g*-PEG on dye adsorption.

loading. This desorption capability of PS-*g*-PEG suggests the possibility that the adsorbent may be used for industrial applications with good reusing capability.

DISCUSSION

Salt Effect

The adsorption can be further facilitated in the presence of salt. The differences in the adsorption capacities in each of the adsorbent systems studied can be explained by the different interactions between the adsorbent and the dye molecules. In the case of F-400, the salt effect on adsorption occurs because the increased salt concentration contributes to neutralization of charges on the surface of the activated carbon and of the dye molecule, which may hinder adsorption.

On the other hand, in the case of PS-*g*-PEG, the salt effects can be explained by the change of reactive sites. Induced dipole-charge interactions between the PEG chain in the PS-*g*-PEG resin and the Direct Blue 78 may explain increased adsorption in the presence of salt as shown in Fig. 8.

As the PEG chain in PS-*g*-PEG is long and flexible, several ethylene glycol units (6–20 units), which are capable of chelating Na^+ ion, could behave as crown ether [Fig. 8; (18)]. Since the Na^+ chelated units exert partially positive

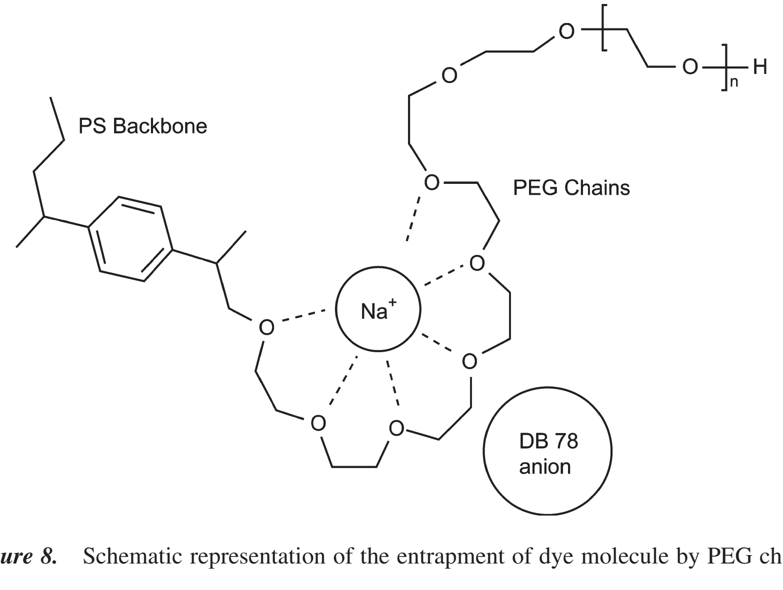


Figure 8. Schematic representation of the entrapment of dye molecule by PEG chain.

charge, they may attract negatively charged dye ions electrostatically. The addition of salt as Na_2SO_4 increases the number of Na^+ chelated units, and thus promotes the electrostatic interaction between the PEG chain and dye particles.

Besides, binding forces between oxygen in PEG chain and the dye molecules may exist through charge-transfer complex formation (15–17). Oxygen in the PEG chain acts as a charge donor, and phenyl ring in dye molecule acts as a charge acceptor. Since Direct Blue 78 molecules have strong electron-withdrawing groups attached to phenyl rings and very long conjugated system, this explanation seems to be plausible. However, the contribution of binding forces, due to charge-transfer complexation to adsorption, is not defined yet. Therefore, additional study is required.

Kinetics

The chemical architecture of the PS-*g*-PEG resin is shown in Fig. 9 (9). In Fig. 9, the bold line represents the PS backbone and thin line represents the grafted PEG chain. Polystyrene is a crosslinked support, and expands its polymeric network by swelling PEG chains. This swelling process exposes the reactive sites, namely, the protruding oxyethylene groups, and makes them accessible to the adsorbate (6). Due to the flexibility of PEG and its large structural dimensions, it may be assumed that a part of the PEG molecule remains on PS framework and the remainder protrudes from PS as loops and tails.

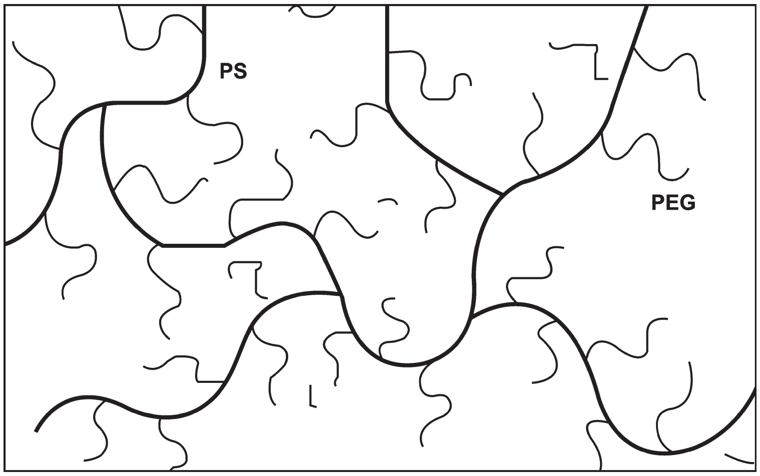


Figure 9. Schematic representation of PS-*g*-PEG resin.

Polystyrene is hydrophobic, and PEG is hydrophilic, therefore, as in the PEG–silica study of Parida and Mishra (18), the oxyethylene groups in PS-*g*-PEG form a hydrophilic interface between a hydrophobic core and an aqueous medium.

The significant contrast in the adsorption kinetics of PS-*g*-PEG and F-400 observed in Fig. 6 can be explained by the difference in the physical structure of the two adsorbents. In F-400, the adsorption sites are limited to the surface of the activated carbon, and time is needed for the dye to diffuse into the surface of pores. On the other hand, in the PS-*g*-PEG resin, the adsorption can occur more easily onto the PEG polymer chains because of its open structure, as described in Fig. 9. The dye can easily diffuse from the bulk phase to the interior space of the polymer matrix, which is relatively accessible due to the high swelling property of PS-*g*-PEG resin.

Desorption

The improved desorption capability of PS-*g*-PEG can also be explained by its physical structure and chemical properties. The ease of desorption is a typical property of physisorption. The well-developed void volume, due to the swelling of PEG chains, enables the eluant to pass through the resin easily, so the dye molecules bound by weak force are eluted without difficulty. Considering the physical structure, the eluant in the carbon system would face the problem of restricted access. This explanation is in accordance with the structural description shown in Fig. 9.

The easy desorption that is indicative of the nature of physisorption and the Langmuir type isotherm that has the nature of a monolayer chemisorption may contradict each other. This phenomenon may come from the unusual spatial structure of the PS-*g*-PEG resin. That is, the PS-*g*-PEG resin has no definite surface and porous structure and the PEG chain is long and flexible as shown in Figs. 8 and 9. Therefore, general explanation for isotherm results may not be applied. The way of DB 78 interaction with PS-*g*-PEG resin is presumed to be through induced dipole-charge interaction that is weak in nature. This gives the isotherm characteristic of monolayer adsorption following Langmuir type since multiplayer interaction is rarely allowed to happen. However, the definitive explanation of the mechanism of desorption on PS-*g*-PEG resin is beyond the scope of this study.

CONCLUSIONS

The dye adsorption properties of PS-*g*-PEG resin, which is a newly synthesized polymer adsorbent, were examined in aqueous solution with a model dye, Direct Blue 78, at various salt concentrations and pHs and compared with that of an activated carbon (F-400). The PS-*g*-PEG resin showed much better adsorption properties than F-400, and its adsorption capacity was substantially influenced by the presence of salt. The dye adsorption on PS-*g*-PEG was rapid, and reached the equilibrium concentrations within 1 hr. In addition, the dye adsorbed on PS-*g*-PEG resin was desorbed easily.

The direct comparison of the sorption capacities of the PS-*g*-PEG resin and F-400 alone cannot imply that the PS-*g*-PEG resin can be an alternative for the commercial activated carbon because of the high cost of synthesis of PS-*g*-PEG resin. The much higher adsorption capacity of PS-*g*-PEG resin over F-400 rather implies that the polymer adsorbent with the specific functional group can be synthesized with the purpose of removing a certain type of dye.

The different adsorption behaviors of the PS-*g*-PEG resin and F-400 were explained in terms of their physical structures and chemical properties. The improved adsorption and desorption capabilities of PS-*g*-PEG resin found in this study suggest the possibility of industrial application.

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