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Separation of Toxic Organotin Compounds from Aqueous Solution by Adsorption

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NOTE

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Abstract

The adsorption characteristics of organotin compound (tributyltin oxide) are established on activated carbons, polymeric adsorbent, and synthetic carbonaceous adsorbents. Maximum adsorptive capacity is obtained with activated carbon containing high percentage of macropores (100–500 Å). For this carbon the adsorptive capacity (g organotin adsorbed/g of adsorbent) at breakpoint is 0.30 and the capacity at exhaustion (with 30 mg/L organotin solution) is 0.50.

Organotin compounds are used as insecticides, fungicides, wood preservatives, acid corrosion inhibitors in chlorinated lubricating oils, and in many other applications (*1*). Fungicidal organotin compounds (such as tributyltin oxide, triphenyltin chloride) have been used as additives to paints against attack by molds, fungi, and marine organisms. The use of these compounds in antifouling hull paints is very effective against some marine organisms. Removal of these paints during overhaul (wet blasting) causes the generation of organotin-contaminated wastewaters. The development of adequate organotin removal techniques from the wastewaters is required in various applications.

The objective of this study is to establish the adsorption characteristics of organotin (tributyltin oxide, $(Bu_3Sn)_2O$, molecular weight 590) on

activated carbons, polymeric adsorbent, and synthetic carbonaceous adsorbents. The adsorption behavior is established by utilizing continuous flow, column systems. The adsorption characteristics of organotin compounds are not reported in the literature. Belfort (*2*) has presented the adsorption behavior of various organic homologs on activated carbon and correlated the molar adsorptive capacity with physicochemical structural parameters. Recently, Martin (*3*) has reported the selection procedure of activated carbon for water and wastewater treatment.

EXPERIMENTAL

The adsorption studies were conducted with a 1.15 cm diameter column, and 25 cm^3 bed volume and flow rates of 9–10 cm^3/min were utilized. The adsorbents used in this study and their properties are shown in Table 1. Tributyltin oxide (TBTO) dissolved in distilled water (concentration 4000–13,000 μg/L as tin) was utilized in this study. Since TBTO is not very soluble in water, concentrations greater than 20,000 μg/L (as tin) could not be utilized. The effluent and influent TBTO concentrations were measured by analyzing tin with a flameless atomic adsorption spectrophotometer. TBTO concentration in μg/L is equal to Sn μg/L $\times$ 2.5.

RESULTS AND DISCUSSIONS

The experimental breakthrough curves for two types of activated carbons are shown in Figs. 1 and 2. Figure 1 shows that Filtrasorb 300 attains breakthrough considerably faster than OL 20 $\times$ 50. In both cases the breakthrough curves are not steep. In order to simulate variations in wastewater characteristics, two column studies were conducted with variable feed TBTO concentrations. The results are shown in Fig. 2. The behavior is quite similar to that of Fig. 1, although the total capacity is somewhat lower than that observed for the constant feed concentration case.

Figure 3 shows the breakthrough curves for the two carbonaceous adsorbents and a synthetic polymeric adsorbent. The quick breakthrough of XE-347 may have been due to the fact that 50 vol-% of pore diameters are less than 6 Å and thus block the entrance of TBTO molecules in the pores. XE-348 is a broader spectrum adsorbent and is most similar to activated carbons. Although both XE-348 and XAD-4 achieved breakthrough at the same time, the total adsorptive capacity of XAD-4 was higher than XE-348.

The adsorptive capacities (in g/g and g/cm^3 adsorbent) of the five adsorbents at breakpoint are computed from Figs. 1 and 3 and shown in

TABLE 1

Adsorbents Used in This Study

	OL 20 × 50	Filtrasorb 300	Amberlite XAD-4	Ambersorb XE-348	Ambersorb XE-347
Type	Activated carbon	Activated carbon	Polymeric adsorbent	Synthetic carbonaceous adsorbent	Synthetic carbonaceous adsorbent
Surface area (m^2/g)	1000–1100	950–1050	725	500	350
Bulk density (g/cm^3)	0.45	0.42	0.70	0.60	0.70
Pore volume (cm^3/g)	0.88	0.85	0.50	0.58	0.41

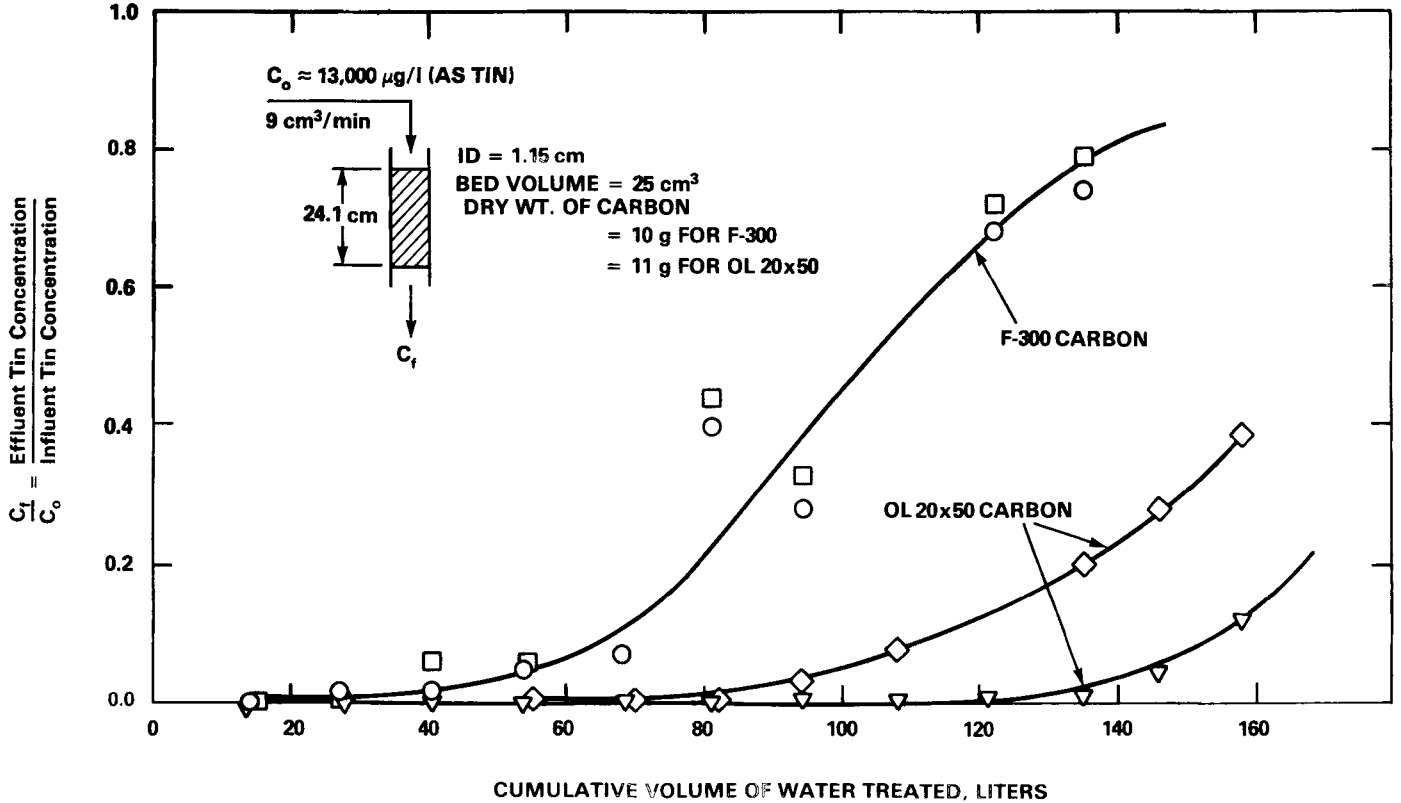

FIG. 1. Breakthrough curves of organotin for two types of activated carbons.

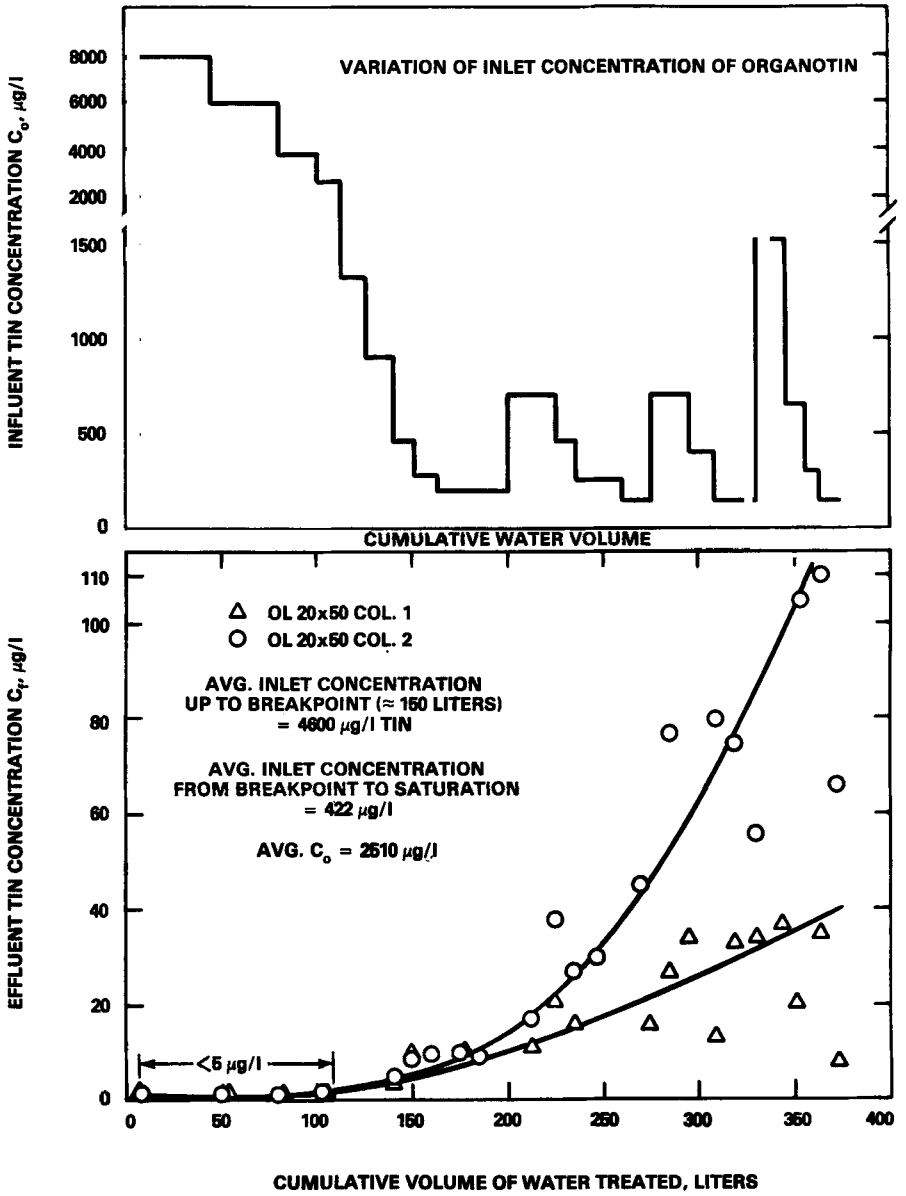

FIG. 2. Breakthrough curves of organotin with variable feed concentration.

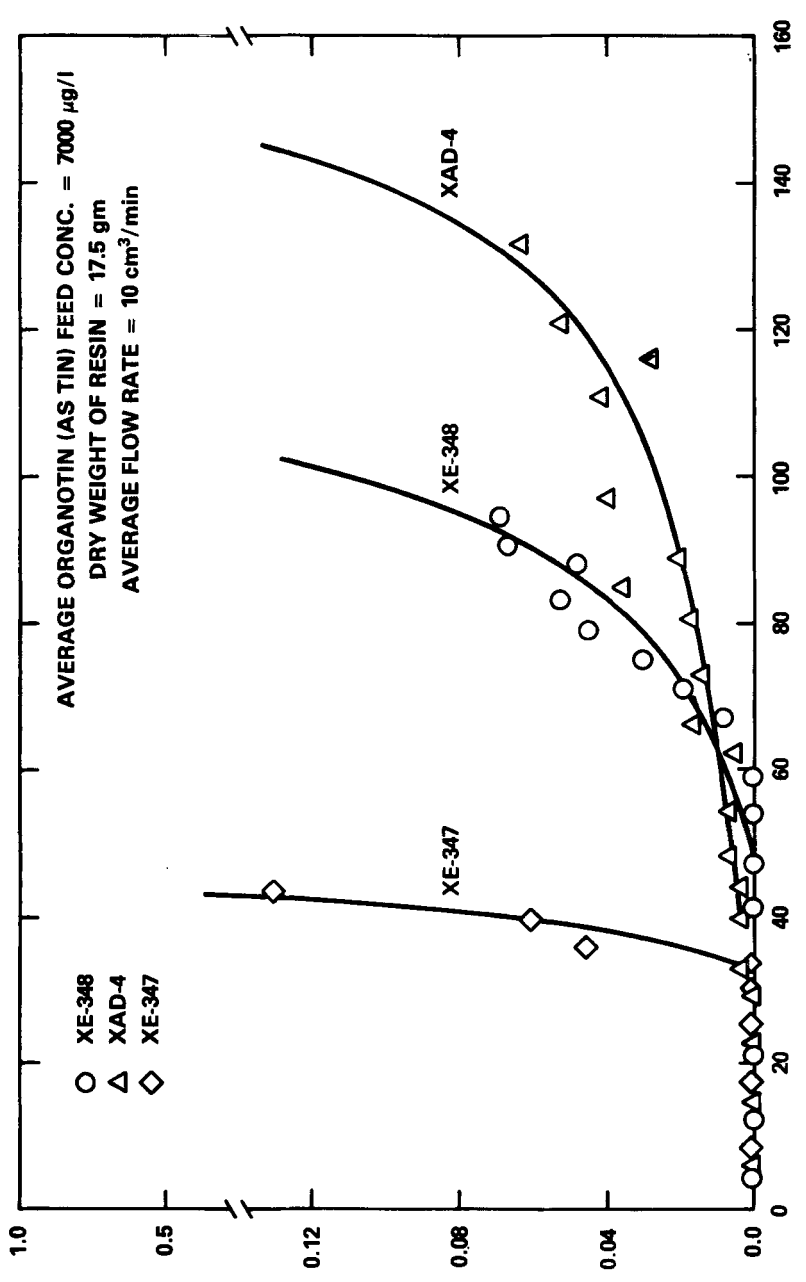

FIG. 3. Breakthrough curves of organotin for carbonaceous and synthetic polymeric adsorbents.

TABLE 2

Comparison of Adsorptive Capacities at Breakpoint (99% removal)

Adsorbent	Gram of organotin adsorbed / Gram of adsorbent	Gram of organotin adsorbed / cm^3 of adsorbent
OL 20 × 50	0.358; 0.250	0.167; 0.118
Filtrasorb 300	0.158	0.063
XE-348	0.065	0.046
XAD-4	0.060	0.042
XE-347	0.035	0.025

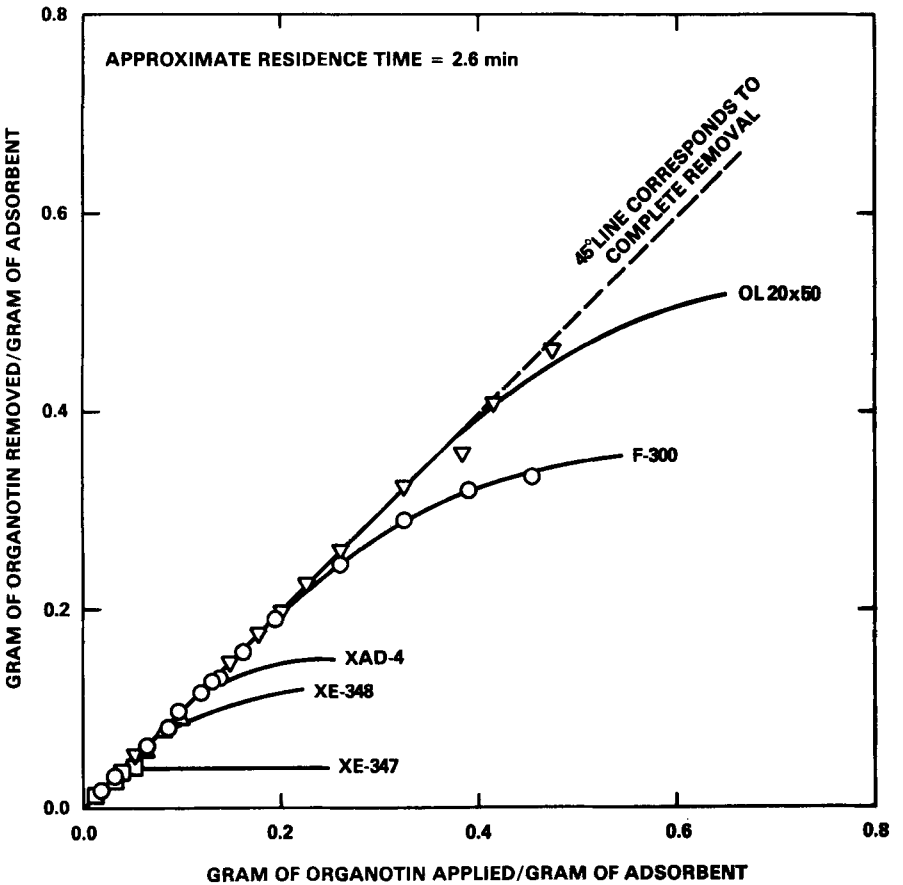

FIG. 4. Relationship between adsorptive capacity and organotin loading for various adsorbents.

TABLE 3

Maximum Adsorptive Capacity at Exhaustion

Adsorbent	Gram of organotin adsorbed / Gram of adsorbent	Gram of organotin adsorbed / cm^3 of adsorbent
OL 20 × 50	0.518; 0.489	0.230; 0.218
Filtrasorb 300	0.358	0.143
XAD-4	0.150	0.105
XE-348	0.110	0.077
XE-347	0.041	0.029

Table 2. The capacity at breakpoint is highest for OL 20 × 50 (which has both micro- and macropores) and lowest for XE-347 (which has very fine micropores).

Since complete exhaustion (except XE-347) curves were not obtained experimentally, the breakthrough curves are extended to the exhaustion point by using an infinite rate mass transfer based adsorption model (*4*). At infinite mass transfer rate the breakthrough curve would be a vertical line. Figure 4 shows the adsorptive capacities as a function of organic loading. Fugure 4 shows that both OL 20 × 50 and Filtrasorb 300 continue to remove organotin even at loadings greater than 0.5 g of organotin applied per gram of adsorbent. Table 3 shows the maximum adsorptive capacities (calculated) of the adsorbents at exhaustion. At exhaustion the adsorbent is in equilibrium with the feed solution. Comparing Tables 2 and 3, it can be observed that at breakpoint the adsorbents are only partially (except XE-347) saturated, thus indicating that series-type column operations would be desirable. It is interesting to observe that the capacity trends agree with the pore volume values shown in Table 1.

The superior adsorptive capacity of OL 20 × 50 activated carbon is due to the presence of a high percentage of macropores (100–500 Å) in the particle structure. The high capacity of this activated carbon would enable long-term operation without the need of any regeneration. Utilizing the throughput volume data from Fig. 1 and the capacities from Tables 2 and 3, it can be easily shown that 76,000 L/d (8 h d) of water containing 1000 μg/L TBTO could be treated in a column (at 16 cm^3/cm^2 min flow) of bed height 60 cm and 5.9×10^5 cm^3 carbon volume (268 kg OL 20 × 50 carbon). The breakthrough concentration of 5 μg/L TBTO would occur after 9300 h of operation. This service time corresponds to only 0.027 kg carbon/h as the usage rate.

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