Regeneration of Carbon Containing Sodium Dodecylbenzene Sulfonate—Cyclic Regeneration with Steam in Fluidized Beds

Cyclic regeneration of activated carbon containing adsorbed sodium dodecylbenzene sulfonate (DBS) was studied in fluidized beds at 1119 K and 101.3 kPa. Optimum restoration of adsorption capacity (97.2% per cycle) resulted when gasification with 21.5% steam was continued for 200 s. At that time, about 85% of the DBS added to the carbon during the adsorption process had been removed. Because of the adsorbed inorganic residue from DBS, these results are somewhat different from earlier studies with the sucrose-carbon system. With sucrose, maximum restoration of adsorption capacity was achieved when a weight equivalent to 100% of the adsorbed sucrose was removed by thermal decomposition and steam gasification.

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SCOPE

The use of activated carbon as an adsorbent has been growing as a result of the increased emphasis on water purity. Economic conditions require regeneration of the spent carbon. The most common regeneration process is a thermal one consisting of three steps: 1) drying of the spent wet carbon; 2) heating to about 1,100 K where thermal decomposition partially restores adsorption capacity; and 3) gasification of residual adsorbed material with an oxidizing gas to restore further capacity. Without the gasification step, continued cyclic use results in build-up of residual material and serious loss in adsorption capacity.

Design of regeneration processes has been handicapped in the past by lack of kinetics data. Recent studies of the kinetics of both the thermal regeneration and gasification steps have provided needed data for several adsorbates (Suzuki et al., 1978; Hashimoto et al., 1979; Chihara et al., 1971, 1981a). When powdered carbon is used, regeneration in fluidized beds has some advantages. Hernandez and Harriott (1976) have used this form of reaction for regeneration studies.

There is little information on conditions for maximum restoration of adsorption capacity when the carbon is used repeatedly in adsorption-regeneration cycles. An important factor is the extent of gasification necessary per cycle to achieve maximum adsorption capacity. This subject has been investigated by Umehara et al. (1982) with sucrose as the adsorbate. Powdered carbon was used and gasification carried out in fluidized beds with steam at 1,151 K and 1 atm pressure. Sucrose leaves only a carbonaceous residue after thermal decomposition, and this residue can be completely gasified with steam. Their results for cyclic operation showed that optimum restoration of adsorption capacity (for sucrose) was obtained when gasification was continued until the particle weight was equivalent to the weight of the original virgin carbon. Probably this particular weight consisted, in part, of sucrose residue since some virgin carbon may have been burned during the gasification process. At 1,151 K the gasification time required was 180 s and 98.5% of the original adsorption capacity was restored after each regeneration.

While sucrose is a pollutant in some wastewaters, it is rather rapidly decomposed by heating (thermal decomposition). More refractory sulfonate salts are also common water contaminants, and these leave an inorganic residue after gasification. To provide cyclic regeneration information on this type of system, data were obtained for sodium dodecylbenzene sulfonate adsorbed on powdered, activated carbon in fluidized beds operated at 1119 K and 1 atm. This system was chosen, in part, because steam-gasification kinetics had been studied in TGA equipment (Umehara et al., 1982a). With such kinetics, weight vs. time results for the gasification process could be predicted for comparison with the results measured in the fluid bed-cyclic regeneration studies.

CONCLUSIONS AND SIGNIFICANCE

Adsorption-regeneration measurements were made in fluidized-bed reactors without removing the carbon particles from the reactors. By using different reactors, each subjected to a specific gasification time, it was possible to determine the effect of extent of gasification on restoration of adsorption capacity for DBS. As might be expected, long gasification times resulted in burning considerable virgin carbon with accompanying loss in adsorption capacity. Very short, or zero, gasification times led to the build-up of inorganic and residual-carbon residues. This also caused a reduction in adsorption capacity. Such reduction was particularly severe, apparently because the inorganic residue blocked active carbon adsorption sites or plugged some of the very small pores in the carbon particles. The reduction caused by inorganic residue was verified by measuring adsorption capacity for DBS on samples of activated carbon that contained previously adsorbed Na_2SO_4, the likely inorganic residue from DBS.

The cyclic regeneration studies were carried out in fluidized beds operated at 1,119 K and 1 atm. For the oxidation (gasification) step, a gas containing 21.5% steam and 78.5% helium was used. At these conditions, maximum restoration of adsorption capacity was achieved when the reduction in weight of adsorbed DBS by thermal decomposition and gasification was about 85% of the weight originally adsorbed. This optimum restoration of adsorption capacity is 97.2% per cycle. The corresponding gasification time is about 200 s.

The kinetics data (obtained in TGA apparatus) for gasification of adsorbed DBS (Umehara et al., 1962a) could be used to predict this observed gasification time with reasonable accuracy.

In comparison with the sucrose system, the rate of gasification of adsorbed DBS is more rapid. This is due to the catalytic activity of the inorganic residue, although thermal decomposition of DBS is slower than decomposition of adsorbed sucrose.

When activated carbon is used repeatedly in adsorption-regeneration steps, it is important economically that maximum adsorption capacity be restored after each regeneration. The regeneration is accomplished by an initial thermal decomposition followed by gasification of the residue with an oxidizing gas. The extend of restoration is determined primarily by the severity of the gasification step. With too little gasification, the build-up of residual carbon reduces adsorption capacity. If too much gasification occurs, capacity is reduced due to loss, by burning, of virgin carbon. We have previously studied this problem (Chihara et al., 1982) with carbon containing adsorbed sucrose, using steam for gasification. Adsorbed sucrose is rapidly decomposed in the thermal step leaving only a carbonacious residue. The results reported here are for a different type of system: Activated carbon containing adsorbed sodium dodecylbenzene sulfonate (DBS). After thermal regeneration and gasification, a nonvolatile sodium salt, probably sodium sulfate, remains. In TGA kinetics studies, Umehara et al. (1982) found that this residue affects significantly the kinetics of regeneration with steam. Since wastewaters may contain both polysaccharides and benzene sulfonates, regeneration information is needed for both types of pollutants.

To establish optimum gasification conditions, it is necessary to follow through several cycles of adsorption and regeneration. To minimize carbon loss, all processes and measurements were carried out without removing the carbon from the reactors. This required special experimental techniques and small-size apparatus so that weighings could be made with the carbon particles in place. Five different reactors were employed to study five gasification times. One cycle consisted of: 1) adsorption and measurement of amount of DBS adsorbed, 2) drying, 3) thermal regenration, and 4) gasification and weighing. Five cycles were carried out in each reactor. Apparatus and procedures are described in the following section.

EXPERIMENTAL

Clear quartz reactors, consisting of a lower conical section and an upper cylindrical tube, were used. The inner diameter at the bottom (entrance) of the conical section was 0.004 m, the diameter of the upper cylindrical section was 0.021 m, with a total length of 0.178 m long. The reactors and the regeneration apparatus (furnace, reactor, constant temperature bath, instrumentation, etc.) were essentially the same as used for the earlier studies with sucrose (Chihara, 1982).

Preliminary Experiments

The same coal-based carbon (type BPL, Pittsburgh Activated Carbon Co.) was used as that employed earlier in the TGA studies (Umehara et al., 1982a). This meant that the rates of gasification observed in our fluidized-bed reactors could be compared with those observed in the TGA apparatus. This is important because bypassing or other forms of imperfect mixing may affect fluidized-bed performance with the result that not all the carbon particles are reacting at the same rate. It was found that gas velocity and mass of carbon in the reactor affected the reaction rate, as evaluated with the assumption of complete mixing. A helium flow rate of 2.5×10^{-6} m^3/s (at 25°C, 1 atm) was observed visually to be sufficient to fluidize the particles and not so large as to cause significant entrainment. With this gas flow, too large a mass of carbon added to the reactor results in rates of gasification less than obtained previously in the TGA apparatus. By trial, it was found that when the sample mass was reduced to 2.0×10^{-4} kg of carbon, the two rates of gasification were essentially the same. In one test, a sample with $q_o = 0.52$ [0.52 kg adsorbed DBS/(kg of virgin carbon)] was first subjected to thermal regeneration at 1,062 K for 240 s. At this stage, 50% of the adsorbed DBS was desorbed. Then the sample was gasified at 1,062 K with a gas containing 2.4% steam and 97.6% helium. By weighing the reactor the gasification rate was determined to be 6.40×10^{-4} kg/(kg of virgin carbon)(s). In the TGA apparatus, Umehara et al. (1982a), at the same temperature, gas composition, and $q_o = 0.52$, found the rate to be 6.43×10^{-4} kg/(kg virgin carbon)(s). As a result of these preliminary experiments, the cyclic regenerations were carried out with a gas rate of 2.5×10^{-6} m^3/s and sample mass in the reactor of about 2×10^{-4} kg.

INITIAL CARBON WEIGHT AND INITIAL DBS ADSORBED FOR EACH REACTOR

Reactor Number (i)	1	2	3	4	5
Steam Reaction Time, s	0	100	200	300	500
Carbon Wt. after Heat Treatment, $W_i(0) \times 10^4$, kg	2.027	2.021	1.979	2.069	1.982
DBS Adsorbed Initially, $A_i(0) \times 10^6$, kg	68.0	67.0	65.0	68.0	67.0
q_o, kg/(kg of Virgin Carbon)	0.335	0.332	0.328	0.329	0.338

Preparation of Reactors

Pretreatment of the virgin carbon consisted of crushing 4 × 8 mesh particles, sieving, and retention of the 100 to 150 mesh fraction. This fraction was then boiled in deionized water to remove dust and dried in an oven at 393 K for 24 hours. Complete properties of the carbon were studied by Chihara et al. (1981); the surface area (BET) is 720×10^3 m^2/kg.

An emply reactor was dried in the furnace (furnace temperature = 1,144 K) for 900 s and weighed. Then 2×10^{-4} kg of the pretreated activated carbon was added to the reactor and the helium flow rate started. With this heat treatment any remaining dust and volatile matter was removed. After heat treatment, the reactor and carbon were again weighed. The virgin carbon weight at this stage, prior to the first adsorption and regeneration cycle, is designated as $W_i(0)$, where i denotes the specific reactor and zero the number of cycles.

Adsorption and Drying

After heat treatment, 20×10^{-6} m^3 of an aqueous solution containing 3,350 ppm of DBS (analytical grade) was added to each reactor, which now contained the sample of carbon particles. The reactors were placed in a constant-temperature (298 K) shaker for 40 hours and then a sample of the aqueous solution was analyzed for DBS. The amount adsorbed $A_i\ (m)$ was calculated from the difference in concentrations of the solutions. Excess solution was removed and the reactor tubes dried in an oven for 24 hours at 423 K.

Regeneration

The reactors were attached to the gas supply and exhaust system with flexible copper tubes. Air was evacuated and replaced with high purity (99.999%) helium. Then the reactor was slowly lowered into a high-temperature furnace and helium flow continued for 240 s. During this period, the particles were heated to 1,119 K. From the earlier TGA experiments (Umehara et al., 1982), it is known that in this thermal decomposition step, 51% of the adsorbed DBS decomposes into volatile products leaving 49% as adsorbed intermediate. The gas stream was then changed to 21.5% steam and 78.5% helium for the gasification step, which was also carried out at 1,119 K. The gasification time, 0 to 500 s, was different in each reactor. These times and other initial conditions of the samples prior to the first regeneration are shown in Table 1.

After steam gasification, the gas flow was changed to pure helium with simultaneous removal of the reactor from the furnace. The helium flow was continued for 300 s and the reactor tube weighed to determine the sample weight $W_i(n)$ and adsorbed residue weight $A_i(n)$ after regeneration.

The foregoing adsorption and regeneration steps were repeated for five cycles in each of the five reactors. The weights during one cycle are shown schematically in Figure 1 for each of the five gasification times (each of the five reactors). The manipulations during adsorption and regeneration are described in more detail by Chihara et al. (1982).

RESULTS OF CYCLIC OPERATION

Thermal Regeneration (Zero Steam Reaction Time)

Figure 2 shows the change in sample (virgin carbon plus adsorbed material) weight with number of cycles for each reactor. The curve for Reactor 1 corresponds to thermal regeneration only. From this curve, after one cycle the weight has increased to 1.164 kg/(kg of initial virgin carbon). This corresponds to an adsorbed residue of about 49% of the original adsorbate. Thus, from Table

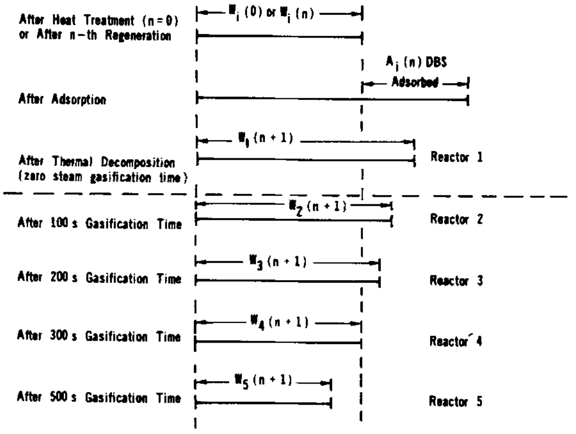

Figure 1. Weight changes in a single cycle.

1, Reactor 1, 49% of 68×10^{-6} is 33×10^{-6} kg per 2.027×10^{-4} kg of virgin carbon or $33 \times 10^{-6}/2.027 \times 10^{-4} = 0.164$. This leads to $W_i(1)/W(0) = 1.164$. Continuing these claculations for subsequent cycles, on the basis that 49% of the adsorbed material remains after thermal regeneration, leads to $W_i(5)/W_i(0) = 1.445$. The observed value in Figure 2 is $W_i(5)/W_i(0) = 1.30$. The lower figure is likely due to the partial solution of the inorganic residue in the aqueous solution used during the adsorption steps. If this residue is Na_2SO_4, it would constitute 20% of the DBS. Using this 20% figure, a value of $W_i(5)/W_i(0) = 1.30$ indicates that about 88% of the inorganic residue dissolves during the adsorption step in each cycle. The dashed curved in Figure 2 for Reactor 1 results from assuming 88% of the inorganic residue dissolves. Actually, this is probably somewhat too high since there is a small loss of sample due to the entrainment while removing the excess DBS solution after adsorption. For comparison, the curve for the sucrose systems (Chihara et al., 1982) is also shown in Figure 2. For sucrose, nearly but not all of the adsorbed material could be removed with only thermal regeneration, in contrast to the DBS system. Since most of the Na_2SO_4 residue is dissolved during the adsorption step, the increase in weight with number of cycles, shown by the curve for Reactor 1 in Figure 2, is due primarily to residual carbonaceous residue. Hence, steam gasification is particularly important with DBS to prevent a build-up in sample weight. This increase in weight results in a loss in adsorption capacity, as shown later.

Effect of Steam Gasification Time

The decrease in sample weight with increasing steam reaction time, Figure 2, is due to the gasification of residual carbonaceous material. For Reactors 4 and 5, the relatively long reaction times resulted in gasification of so much virgin carbon that the sample weight actually decreased with number of cycles. For Reactor 3, the increase in weight due to accumulation of undissolved inorganic material is nearly balanced by the gasification of virgin carbon and carbonaceous residue.

OPTIMUM GASIFICATION

The curves in Figure 2 for different reactors suggest that there is an optimum steam-reaction time for restoration of adsorption capacity. For design purposes, it is desirable to predict this optimum from the kinetics of the steam reaction. Such kinetics data and the corresponding rate equation have been determined previously (Umehara et al., 1982a) from TGA measurements. The first step in *a priori* design is to use the rate equation to predict the sample weight change with time. Then the adsorption capacity data, obtained in the present study, can be related to the sample weight to establish the optimum gasification time.

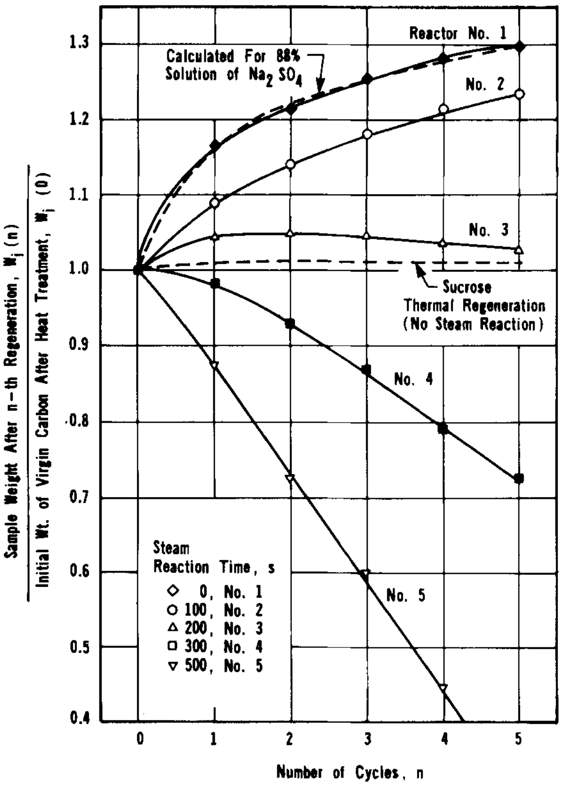

Figure 2. Sample weight change with number of cycles.

Predicted Gasification Results (Sample Weights)

It has been found (Umehara et al., 1982a) that increasing the amount of adsorbed DBS (that is, q_o) increases the steam gasification rate, and that this is due to the catalytic effect of the inorganic adsorbed material. Data for three levels of q_o and at three temperatures, as obtained in the TGA apparatus, were used to determine, empirically, that the rate was proportional to $q_o^{2.8}$. Adding this effect to the rate equation for gasification, as established by Umehara et al. (1982a), yields

$$R = \frac{2.82 \times 10^5 \exp(-1.79 \times 10^5/R_gT)\, q_{\text{inorg}}^{2.80}\, p_{H_2O}}{1 + k_2 p_{H_2} + k_3 p_{H_2O}} \tag{1}$$

where

$$k_2 = 1.02 \times 10^2 \exp(1.70 \times 10^5/R_gT),\ \text{Pa}^{-1}$$

$$k_3 = 1.18 \times 10^{-3},\ \text{Pa}^{-1}$$

$$R = \text{kg gasified/(kg virgin carbon)(s)}$$

In Eq. 1, q_{inorg} is taken as 0.20 of q_o. The empirical function $q_{\text{inorg}}^{2.8}$ is not likely to be valid over a wide range of q_o values.

Figure 3 shows predicted (Eq. 1) and experimental changes in sample weight per cycle vs. gasification time. The results are for $T = 1{,}119$ K, 21.5% steam and 79.5% helium. The uppermost (solid) curve for the change in weight for the first cycle is a slightly higher than observed (line through triangular points). This is because there is a very small amount of *thermal* decomposition at temperatures between 748 and 1,119 K. This loss of weight is not accounted for by Eq. 1.

After the first regeneration, 88% of the residual inorganic residue dissolves during the readsorption step, according to the earlier calculations. Using this result, and assuming that the adsorption capacity after each cycle is the same as the initial capacity, the lower dashed curve is that predicted by Eq. 1. This curve applies for each cycle, 2 to 5, since adsorption capacity is assumed to be the same for each of those cycles. The predicted and experimental results agree reasonably well except at zero gasification time. For this extreme case there is a large build up of inorganic residue and the readsorption capacity is greatly reduced, Figure 4 (Reactor 1).

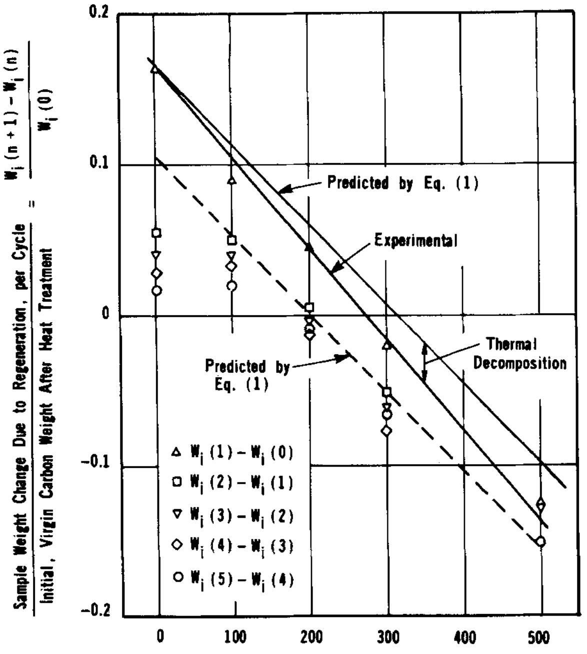

Figure 3. Predicted and experimental steam gasification results for $T = 1{,}119$ K and 21.5% steam.

Then the assumption of constant adsorption capacity is not correct. This probably explains the large and increasing (with number of cycles) deviation between predicted and experimental data in Figure 3 at zero time.

Adsorption Capacity vs. Number of Cycles

The adsorption capacity for DBS is a direct measure of the efficiency of regeneration. Figures 4–6 display this efficiency in different ways. Figure 4 shows the amount adsorbed, $A_i(n)$, divided by the initial adsorption capacity, $A_i(0)$, vs. the number of cycles. The loss in capacity is very large for Reactor 1, corresponding to zero steam gasification time. This result shows that the residual adsorbed material (inorganic plus carbonaceous residue) which builds up with each cycle for Reactor 1 adversely affects the adsorption capacity of the virgin carbon. The adsorption capacity for Reactor 5 also decreases with the number of cycles. This is due to the loss of virgin carbon by gassification, which occurs at long gasification times. Maximum retention of adsorption capacity is for Reactor 3 for which the gasification time is sufficient to remove most of the residual carbonaceous material, but not long enough to gasify a significant amount of virgin carbon.

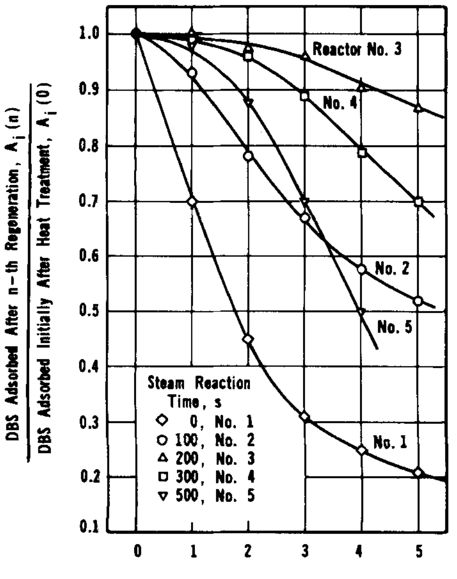

Figure 4. Effect of cycling on DBS adsorption capacity ($T = 1{,}119$ K, 21.5% steam).

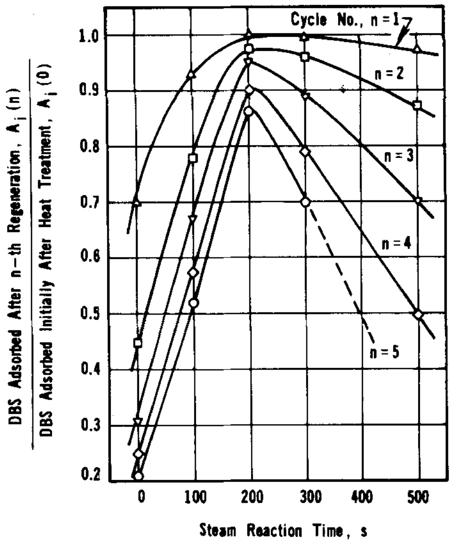

Figure 5. Effect of steam reaction time on DBS adsorption capacity.

This conclusion is more clearly shown in Figure 5 where the fraction of initial adsorption capacity is plotted vs. steam reaction time. After five cycles the loss in capacity is least for 200 s reaction time (Reactor 3) and is about 13.5%. This corresponds to a restoration of adsorption capacity of 92.7% per cycle.

Figure 6 relates the fractional adsorption capacity to the sample weight, $W_i(n)$. It is this relationship which can be used with the kinetics data (Eq. 1) to determine optimum regeneration conditions. While the optimum is not well defined, it corresponds approximately to a sample weight of about $W_i(n)/W_i(0) = 1.05$. Since the initial value, Table 1, is $q_o + 1 = 1.335$, optimum regeneration is achieved when $(1.335 - 1.05)/(1.335 - 1.0)$, or 85% of the weight of the DBS adsorbed, is removed by thermal and steam gasification. This result applies for thermal decomposition up to 1,119 K and gasification at that temperature. The amount removed for optimum restoration of adsorption capacity might be somewhat different at different temperatures and certainly for different adsorbates. For example, the dashed lines in Figure 6 show results for sucrose adsorbed on the same activated carbon

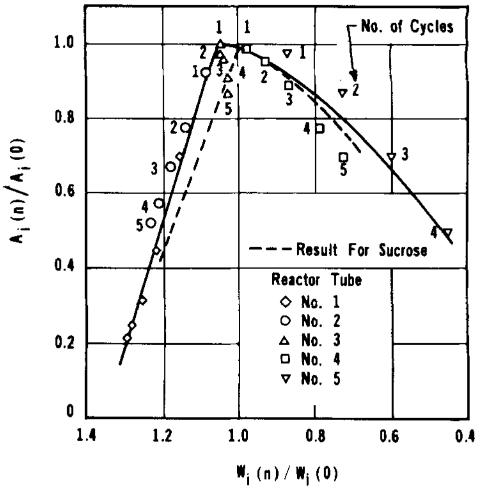

Figure 6. Relation between adsorption capacity and sample weight.

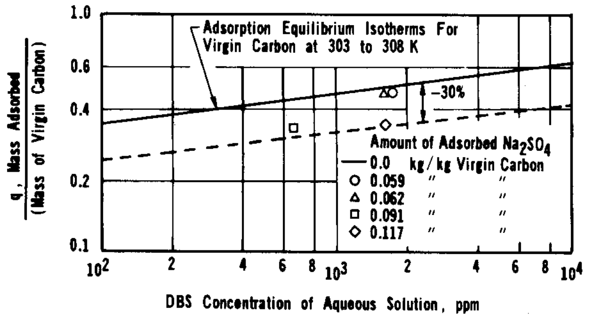

Figure 7. DBS absorption equilibrium data for adsorbed Na_2SO_4 samples and for virgin carbon.

(Chihara et al., 1982). In this case the optimum occurs when $W_i(n)/W_i(0)$ is approximately equal to 1.0; that is, when the gasification is continued until a sample weight corresponding to complete removal of the adsorbed material is achieved. It should be noted that the difference between the systems is not due substantially to the inorganic residue with DBS, because 88% of this is dissolved during the readsorption step. The steeper left sides of the curves in Figures 5 and 6 show again that leaving a residue on the activated carbon, due to insufficient gasification, has a more adverse effect on adsorption capacity than gasifying virgin carbon. In other words, it is better to err on the side of excessive gasification.

In designing a regeneration system for optimum restoration of adsorption capacity, the weight loss during gasification as calculated from Eq. 1 should be about 85% of the weight of the adsorbed DBS. This corresponds approximately to the removal of the organic part of DBS. The time required at 1,119 K would be about 200 s.

EFFECT OF Na_2SO_4 ON ADSORPTION CAPACITY

As noted earlier, the data in Figure 4 suggest that inorganic residue reduces the adsorption capacity of activated carbon for DBS. To study this situation further, adsorption capacities were measured for activated carbon on which pure Na_2SO_4 had been adsorbed. The Na_2SO_4 pretreatment was carried out by adding an aqueous solution of reagent grade Na_2SO_4 (concentration = 1.3×10^5 ppm) to flasks containing virgin carbon particles, shaking at 298 K for 40 hours, filtering, and then drying for 24 hours at 423 K. The amount of adsorbed Na_2SO_4 was determined by weighing. Then, the adsorption capacity for DBS was determined in the same manner.

The results are shown in Figure 7 where the solid line represents the DBS adsorption capacity for virgin activated carbon, as determined earlier (Umehara et al., 1982). The open points show the effect of adding Na_2SO_4. For low adsorbed concentration of Na_2SO_4 (0.059 and 0.062 kg/kg), the reduction in DBS adsorption capacity is but a few percent. However, when the Na_2SO_4 adsorbed reaches 0.117 kg/(kg of virgin carbon), the DBS capacity is reduced by 30%. This is in agreement with the sharp reduction in capacity seen in Figure 4 for Reactor 1. It seems reasonable to postulate that the inorganic residue from DBS, like Na_2SO_4, adversely affects the adsorption capacity of activated carbon. This could occur by covering active adsorption sites of the carbon or by plugging some of the very small pores.

ACKNOWLEDGMENT

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NOTATION

$A_i(n)$ = DBS adsorbed after nth regeneration, kg
i = number of reactor tube
k_2, k_3 = parameters in rate equation, Eq. 1, Pa^{-1}
n = number of regenerations; $n = 0$ indicates initial condition after heat treatment
P_{H_2}, P_{H_2O} = partial pressure of hydrogen and steam, Pa
q = DBS adsorbed per unit mass of virgin carbon; q_o = DBS adsorbed initially per unit mass of virgin carbon
$q_{Na_2SO_4}$ = mass of Na_2SO_4, adsorbed per unit mass of virgin carbon
q_{inorg} = mass of adsorbed inorganic residue per unit mass of virgin carbon
R = rate of steam gasification, kg/(kg of virgin unburned carbon)(s)
R_g = gas constant, J/(mol)(K)
T = temperature, K
$W_i(n)$ = mass of sample in reactor i after nth regeneration, kg; $W_j(0)$ = mass of sample (virgin carbon) after heat treatment, but before first adsorption

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