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Powdered carbon improves activated sludge treatment

Process provides alternate to granular activated carbon tertiary treatment of wastewater

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Use of powdered activated carbon is an attractive approach for improving refinery activated sludge effluent. Its use is a viable alternative to granular activated carbon tertiary treatment for meeting proposed 1983 Best Available Technology Economically Available (BATEA) effluent quality standards as required by the Environmental Protection Agency (EPA).

The proposed process involves adding powdered activated carbon to the aeration tank of the activated sludge process, achieving cost effectiveness by operating at a very high sludge age and a low carbon dose. Effective removal of oil and colloidal solids in the pretreatment step is necessary for successful operation.

Effluent quality depends upon both the equilibrium mixed-liquor carbon concentration and the surface area of the carbon. An experimental carbon with a high surface area appears to be several times more effective than the best commercial carbons in achieving an effluent quality standard. Pore size of the activated carbon has no apparent effect upon effluent quality.

In general, the process can be used to meet only the long-term average effluent quality proposed for BATEA. Daily maximum and 30-day maximum variability goals, as presently defined, cannot be met.

The proposed process also enhances nitrification at low temperatures and dampens effects of increased hydraulic flow rate on the activated sludge process. Both phenomena will help to decrease effluent variability.

According to the EPA guidelines for treating refinery wastewaters,[1] the sequence shown in Fig. 1 is recommended for current Best Practical Technology Currently Available standards. For meeting 1983 BATEA goals the guidelines recommend an add-on process using granular carbon adsorption. However, this approach may be both inefficient and very costly. So far as is known, its effectiveness has never been adequately demonstrated. Moreover, preliminary estimates indicate that capital and operating costs for the granular carbon adsorption and

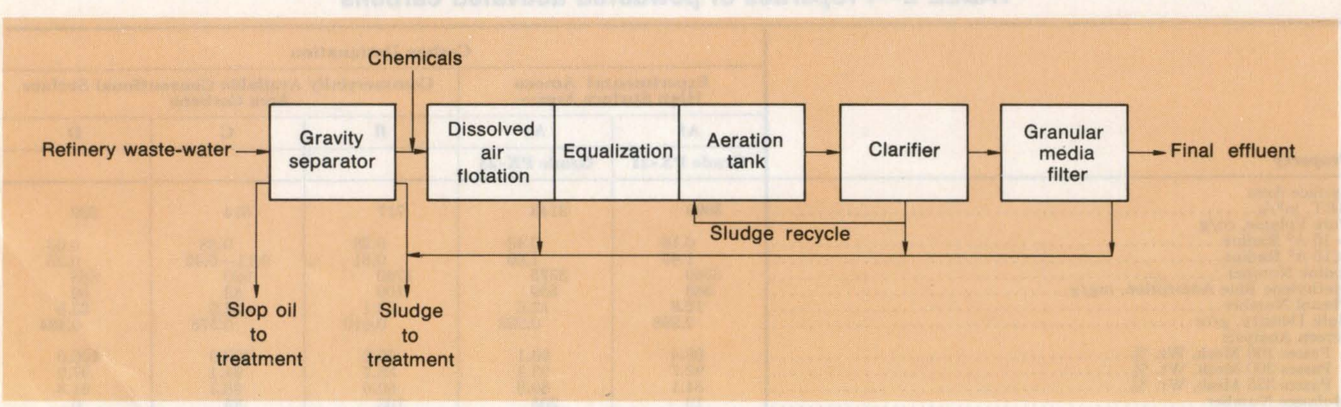

Fig. 1—Simplified refinery Best Practical Technology wastewater treatment system.

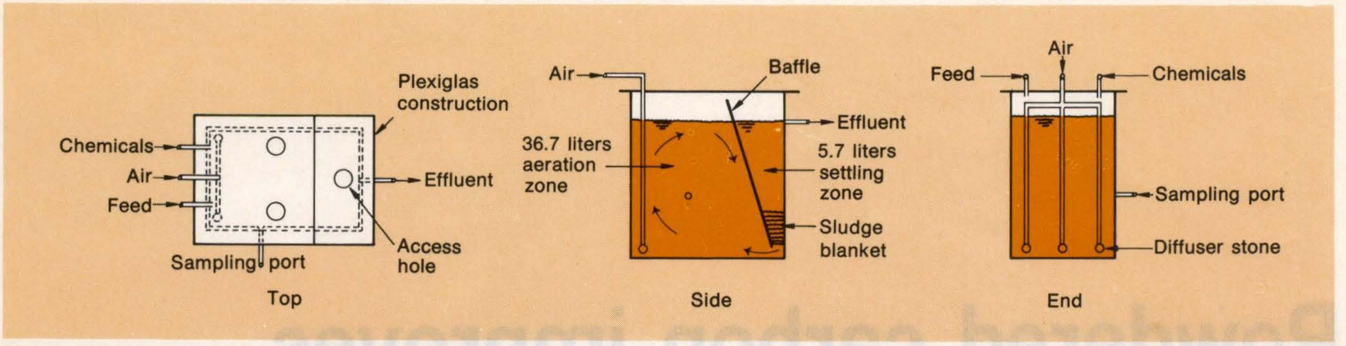

Fig. 2—Activated sludge reactor used in pilot work.

regeneration facilities may equal or exceed those of the entire current activated sludge process.

By contrast, both patents and research studies[2-25] indicate that powdered activated carbon may be a practical and economical substitute for granular carbon. For example, powdered carbon costs only about one-half as much as granular—\$0.65/kg versus \$1.20/kg.[15] In addition, recent studies have shown that powdered carbon can be added directly to the mixed-liquor in activated sludge aeration tanks.[21, 22, 23, 24] Thus, appropriate alterations in operating procedures may eliminate the need for regeneration by making it economically feasible to discard the spent carbon with the waste sludge.

TABLE 1—Pilot plant operating conditions

Aeration Zone Volume	36.7	liters
Settling Zone Volume	5.7	liters
Nominal Flow Rate	2.45	liters/hr.
Nominal Hydraulic Retention Time	15.0	hours
Nominal Settling Time	2.33	hours
Air Flow Rate	300	liters/hr.
pH	6—8.5	
Caustic Addition Rate	0.12—0.30	liters/hr.
Phosphorous Added to Feed	3	mg/liter
Temperature	Ambient (4—31°C)	

Analytical Work

Frequency	Analysis Performed[27, 28]
Daily	Influent and mixed-liquor pH, temperature, influent flow rate, caustic addition rate. Carbon addition and sludge wastage.
3 Times a Week	Influent and effluent total and volatile suspended solids, soluble organic carbon, soluble chemical oxygen demand, soluble ammonia nitrogen, and soluble phenolics. Mixed-liquor suspended solids and mixed-liquor volatile suspended solids. Sludge volume index.
Once a Week	Material balances to calculate quantity of sludge to be wasted to maintain desired sludge age.

In general, the cost effectiveness of a powdered carbon process increases with the concentration of carbon maintained in the mixed-liquor. A mass balance of such a process is represented by the following equation:

$$C = \frac{C_i \, \Theta_c}{\Theta_h} \tag{1}$$

where

C = Equilibrium mixed-liquor carbon concentration (mg/1)
C_i = Influent carbon concentration (mg/1)
Θ_c = Sludge age (days)
Θ_h = Hydraulic retention time in the aeration tank (days)

Equation 1 shows that the equilibrium mixed-liquor carbon concentration is proportional to the product of the influent carbon concentration (carbon dose) and the sludge age. Thus, equilibrium carbon concentration can be increased by increasing the carbon dose, or the sludge age or both. Therefore, to keep carbon costs to a minimum, it is desirable to operate at as high a sludge age as possible and not at an excessively long hydraulic retention time.

A possible drawback to operation at a high sludge age is the increased risk that toxic, inhibitory, or inert materials will build up in the aeration tank. For example, a build-up of oily solids could reduce the oxygen transfer efficiency and inhibit both the nitrifying and organic carbon utilizing organisms. The dissolved oxygen concentration in the mixed-liquor could also become too low for

TABLE 2—Properties of powdered activated carbons

Property	Carbon Designation				
	Experimental Amoco High Surface Area—		Commercially Available Conventional Surface Area Carbons		
	A1 Grade PX-21	A2 Grade PX-23	B	C	D
Surface Area					
BET, m²/g	3099	3148	717	514	532
Pore Volume, cc/g					
> 15 A° Radius	0.16	0.43	0.28	0.38	0.03
<15 A° Radius	1.45	1.60	0.51	0.11—0.42	0.25
Iodine Number	3349	3375	1790	920	888
Methylene Blue Adsorption, mg/g	586	550	100	83	50
Phenol Number	12.8	12.6	34.1	22.9	23.8
Bulk Density, g/cc	0.298	0.228	0.610	0.576	0.484
Screen Analysis					
Passes 100 Mesh, Wt. %	98.4	99.1	99.2	100.0	100.0
Passes 200 Mesh, Wt. %	92.7	93.4	86.7	94.4	97.9
Passes 325 Mesh, Wt. %	84.1	80.8	60.6	68.3	91.8
Molasses Number	10	205	103	85	0

effective nitrification, and the final clarifiers could become overloaded. Therefore, it is desirable in the pretreatment step to remove as much solid material as possible from the wastewater before it enters the aeration tank.

To evaluate the effects of such variables in a process using powdered carbon, an extensive 15-month four-phase pilot plant study was carried out at Amoco Oil Company's Texas City refinery. Pilot plants operating in parallel with the refinery activated sludge process facility were fed the same wastewater for treatment. Specific variables investigated were:

- Carbon type, including surface area and pore volume
- Carbon addition rate
- Sludge age
- Pretreatment of feed to remove oil and solids.

EXPERIMENTAL EQUIPMENT

Fig. 2 shows the configuration of the pilot plants. Each had a volume of 42 liters, and as many as eight units were operated in parallel during portions of the study. They were housed in a rain-tight enclosure but were neither heated nor cooled. Thus, the temperature of the mixed-liquor varied from 4° C to 31° C.

Operating conditions and analytical procedures are summarized in Table 1. The pH was checked daily and controlled by addition of caustic at a constant rate. Dibasic potassium phosphate, K_2HPO_4, was added to satisfy the phosphorus requirement of the microorganisms.

The wastewater feed, a slipstream from the pressure filters of the refinery treatment plant, was passed through a pilot gravity sand filter before being fed to the pilot plants.

Table 2 summarizes the characteristics of the five powdered carbons evaluated. Amoco's experimental high-surface-area carbons are designated as A1 and A2, PX-21 and PX-23, respectively. Those designated as B, C, and D are commercially available carbons having a much lower surface area. Carbon A2 (PX-23) has the highest pore volume.

Effectiveness was judged on the basis of effluent standards proposed for a BATEA facility[1] (Table 3).

TABLE 3

	Concentration mg/liter
Total Organic Carbon (TOC)	15
Chemical Oxygen Demand (COD)	24
Ammonia (NH_3-N)	6.3
Phenolics	0.02

These standards are for a Class C refinery and are based on the guideline effluent flow rate of 0.46 m^3/m^3 of crude thruput per stream day (19 gal/bbl.). Because the BATEA treatment sequence will undoubtedly result in very low concentrations of effluent suspended solids, only the soluble components of the effluent were measured.

To obtain high sludge ages, effluent suspended solids were allowed to settle in 30-gallon plastic containers and then were returned to the pilot plants periodically. At any given sludge age, all plants were allowed to reach steady-state operation over an extended period of time. Then performance data were taken over a 30-day period.

RESULTS

The four phases of the study were carried out in sequence, with the design of succeeding phases based on the results of the preceding ones (Table 4).

TABLE 4

Phase	Objective
I	Effect of carbon type at an addition rate of 100 mg/liter and a sludge age of 20 days with prefiltered feed.
II	Effect of carbon type at an addition rate of 200 mg/liter and a sludge age of 20 days with prefiltered feed.
III	Effect of increasing sludge age to 60 days and reducing carbon addition rate to 25 mg/liter with unfiltered and prefiltered feed.
IV	Effect of further increasing sludge age to 150 days while reducing carbon addition rate to 10 mg/liter.

Phases I and II. The results of Phases I and II (Table 5) indicate that powdered activated carbon significantly enhances the performance of a refinery activated sludge process. Improvement in the quality of the effluents from carbon-fed plants ranged from 65% for soluble organic carbon up to 95% for phenolics. At the 200 mg/liter addition rate, the results usually satisfied the BATEA effluent quality goals. The high surface area carbon A1 was significantly more effective than the other three. The commercially available carbon B produced slightly better effluent than carbon C, which would be expected if efficiency is proportional to surface area. Because nitrification was essentially complete in the control unit, carbon addition could not improve ammonia conversion. Carbon D, which is derived from wood charcoal and has a

TABLE 5—Phases I and II—Effect of carbon type and addition rate on effluent quality* 50% probability data during 30 days of steady-state operation sludge age = 20 days

	Concentration, mg/liter					
		Pilot Plant Effluent				
Component	Filtered Influent	No Carbon	Carbon A1	Carbon B	Carbon C	Carbon D
Phase I: Carbon Addition Rate = 100 mg/liter Equil. Mixed-Liquor Temp = 31°C, Carbon Conc = 3200 mg/liter						
SOC	72.0	22.0	12.5	17.5	18.5	23.0
SCOD	230	73	28.5	48	44	65
NH_3-N	25.8	0.5	0.2	0.5	0.5	0.5
Phenolics	4.35	0.018	0.003	0.010	0.010	0.017
Phase II: Carbon Addition Rate = 200 mg/liter Equil. Mixed-Liquor Temp = 25°C, Carbon Conc = 6400 mg/liter						
SOC	70.0	26.5	9	13.5	15.5	.....
SCOD	230	58	17	24	28	.....
NH_3-N	25.4	0.2	0.2	0.2	0.1	.....
Phenolics	4.06	0.020	0.001	0.001	0.003	.....

*BATEA effluent standards in mg/liter are:

Soluble Organic Carbon (SOC)	15
Soluble COD (SCOD)	24
Ammonia Nitrogen (NH_3-N)	6.3
Phenolics	0.02

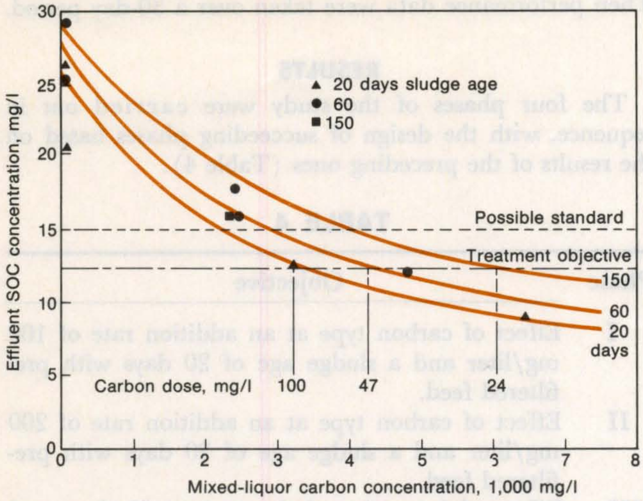

Fig. 3—Effect of mixed-liquor carbon concentration on effluent soluble organic carbon.

significantly lower pore volume than the others, performed so poorly in Phase I that it was dropped from further consideration. The performance of carbon A1 at 100 mg/liter dose was about as effective as carbon B at 200 mg/liter, or about twice as effective as the best commercially available carbon tested.

Phase III. Table 6 shows the effects of sludge age and feed filtration upon performance. The plant with filtered feed performed better than one with unfiltered feed, and a sludge age of 60 days was better than one of 20 days. No deterioration in the settling characteristics of the mixed-liquor suspended solids was observed at this higher sludge age.

At a sludge age of 20 days the plant with filtered feed performed marginally better than the one with unfiltered feed. Undoubtedly, greater differences in effluent quality would have been observed in a plant operated at a sludge age of 60 days with unfiltered feed. (Not recorded in these data, however, is the complete failure of the plant fed unfiltered feed shortly after cessation of data gathering for this steady-state period.)

Table 6 also shows how pore size and surface area affect the performance of the carbons. Carbons A1 and A2 have approximately the same surface area, but carbon A2 has much larger pores. Yet, at an equivalent addition rate of 50 mg/liter, both carbons showed about the same performance. Thus, large pore diameters are not required for effective treatment of this refinery wastewater. Moreover, plants fed 50 mg/liter of either A1 or A2 performed much better than the plant fed 100 mg/liter of carbon B. In fact, these high-surface-area carbons are between two and four times more effective than carbon B in enhancing SOC and soluble COD removal.

A comparison of the data in Tables 5 and 6 shows that a low carbon dose and a high sludge age enhance an activated sludge process almost as much as do a high carbon dose and a low sludge age.

It is possible that the difference in performance is solely due to difference in temperature between the phases—mean operating temperature during Phase III was only 14° C, whereas during Phases I and II temperature averaged 31° C and 25° C, respectively.

Also observed during the lower operating temperature of Phase III was an increase in the ammonia removal efficiency of the carbon-fed pilot plants. This phenomenon was unexpected because activated carbon does not normally adsorb ammonia. Possibly, the increased removal rate is due to the adsorption of potentially toxic or inhibitory organic materials which would reduce the rate of nitrification if left in solution. The control plant in Phases I and II had little difficulty in achieving full nitrification, perhaps because of the higher temperature.

Phase IV. As shown in Table 7, Phase IV was designed to push the activated sludge system to the limit by increasing sludge age to 150 days and decreasing carbon addition to 10 mg/liter. Further, in one of the plants, hydraulic retention time was reduced to 7.5 hours, compared with 15 hours in the other plants.

Despite similarities in influent quality during all four

TABLE 6—Phase III—Effect of carbon type and addition rate, sludge age, and influent pretreatment on effluent quality
50% probability data during 30 days of steady-state operation equil. mixed-liquor temp = 14° C, carbon conc. = 2400 mg/liter

	Carbon		Influent Concentration, mg/liter			
Influent Pretreatment	Type	Addition Rate, mg/liter	SOC	SCOD	NH_3-N	Phenolics
Filtered Feed...	—	—	73.5	294.5	19.3	3.95
			Effluent Concentration, mg/liter			
			Sludge Age = 20 Days			
Unfiltered......	—	—	32.0	103.5	12.1	0.027
Filtered........	—	—	29.0	83.0	14.5	0.027
			Sludge Age = 60 Days			
Filtered........	—	—	25.0	65.9	5.1	0.019
Filtered........	B	100	16.0	40.3	0.2	0.001
Filtered........	A1	50	12.0	27.5	0.1	0.002
Filtered........	A1	25	16.0	50.3	0.4	0.006
Filtered........	A2	50	13.0	31.0	1.8	0.004

TABLE 7—Phase IV—Effect of high sludge age, low carbon addition rate, and decreased hydraulic retention time on effluent quality*
50% probability data during 30 days of steady-state operation, equil. mixed-liquor temp = 27° C

Carbon					Effluent Conc, mg/liter			
Type	Addition Rate, mg/liter	Sludge Age, days	Hydraulic Retention Time, hr	Equil. Mixed Liquor Carbon Conc, mg/liter	SOC	SCOD	NH_3-N	Phenolics
—	—	60	15	—	29	99	0.1	0.018
B	25	60	15	2400	22	64	0.1	0.010
A1	25	60	15	2400	18	52	0.1	0.010
A1	25	60	7.5	4800	17	46	0.3	0.010
A1	10	150	15	2400	16	49	0.1	0.010

*Filtered influent contained 78 mg/liter SOC, 270 mg/liter SCOD, 29 mg/liter NH_3-N, and 3.25 mg/liter phenolics.

phases, during Phase IV the effluent SOC and COD of the control increased by about 30-35% over that observed during the first three phases, despite a mean temperature of 27° C (*c.f.* 14° C during Phase III). All pilot plants essentially nitrified completely.

Remarkably, however, the plant with 10 mg/liter of high surface area carbon A1 at a sludge age of 150 days produced an effluent whose soluble organic carbon concentration was 50% lower than that of the control reactor and slightly lower than that of all of the other pilot plants. The plant dosed with 25 mg/liter carbon A1, with one-half the hydraulic capacity of the other plants, produced the second best effluent.

The outstanding performance at a sludge age of 150 days indicates that refinery activated sludge processes can be operated with very little added carbon. The dose may be low enough so that the carbon need not be regenerated but be discarded with the waste activated sludge. At a very high sludge age, there will be smaller quantities of waste sludge to be disposed of.

The data in Table 7 also indicate that powdered carbon can be used to increase the hydraulic capacity of an activated sludge plant, as proposed by others,[13] or to increase the effluent quality of an overloaded plant. The carbon-fed plant that operated at one-half the hydraulic retention time of the control produced an effluent 50% better than that of the control. Experience with pilot activated sludge plants operated at several of Amoco's other refineries has shown that conventional activated sludge processes cannot be operated successfully with a hydraulic residence time of only 7½ hours.

STATUS

The data from Phase IV indicate that the limits of the powdered carbon enhanced activated sludge process have not been reached. In addition, more data are needed before economic studies can be made to weigh the possible options for achieving a given effluent quality:

- High fresh carbon dose at moderate sludge age (20-60 days) with regeneration of spent carbon;

- Low fresh carbon dose at high sludge age (60-150 days) with no regeneration of spent carbon.

Cost analyses should be made for each of these extreme options, and several intermediate ones, and compared with those for tertiary treatment with granular carbon technology.

Fig. 3 shows the qualitative curves this pilot study has generated. Of course, the one for the 150-day sludge age is purely speculative because only one data point exists. However, the trend of the data does show that effluent quality is a function of mixed-liquor carbon concentration. The curves are probably asymptotic to a residual organic carbon concentration, but over the range investigated an increase in mixed-liquor carbon concentration causes a decrease in effluent soluble organic carbon. Furthermore, the relationship between effluent quality, sludge age and carbon dose is clearly non-linear. For example, to achieve an effluent quality of 12.5 mg/liter of soluble organic carbon, the three options are: 100 mg/liter of carbon at a sludge age of 20 days; 47 mg/liter of carbon at a sludge age of 60 days; 24 mg/liter

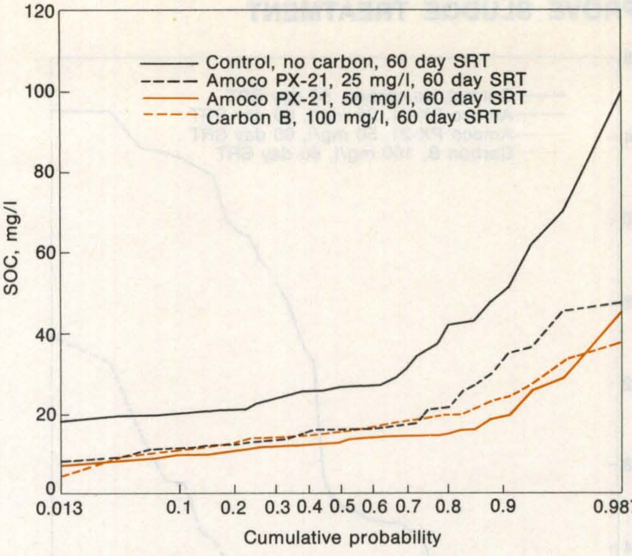

Fig. 4—Soluble organic carbon—Phase III.

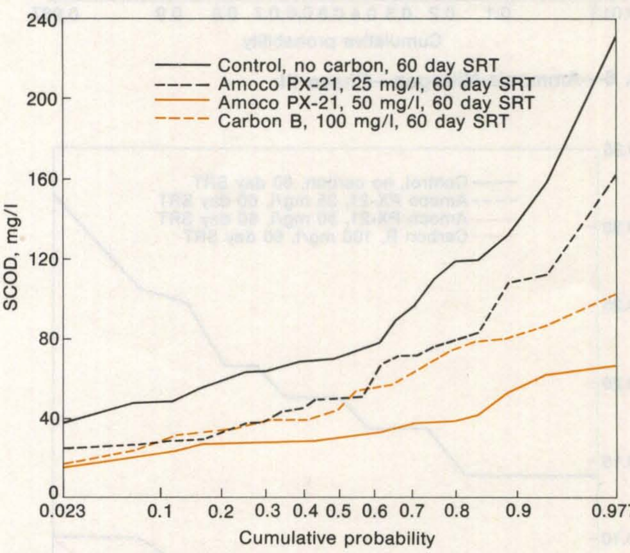

Fig. 5—Soluble chemical oxygen demand—Phase III.

of carbon at a sludge age of 150 days. If the relationship were linear, the values calculated from a base case of 100 mg/liter at a 20-day sludge age would be 33 mg/liter and 13 mg/liter at 60 days and 150 days, respectively.

Apparently, the process loses effectiveness because of incomplete microbial regeneration. Microbial regeneration of the spent carbon is probably not as effective as using fresh carbon; some materials adsorbed by the carbon are undoubtedly non-biodegradable, even after 150 days of contact with microorganisms in the pilot plant. The ability to retain significant effectiveness even at 150 days is the key to cost effective high sludge age operation with powdered activated carbon. Of course, there may be other reasons why carbon loses effectiveness at high sludge age, such as production of cell lysis products which are then adsorbed by the carbon.

EFFLUENT VARIABILITY

Variation in effluent quality over a 30-day (or longer) period is extremely important. The EPA[1] has set the daily maximum variability equivalent to the 99% probability value and the 30-day maximum variability to

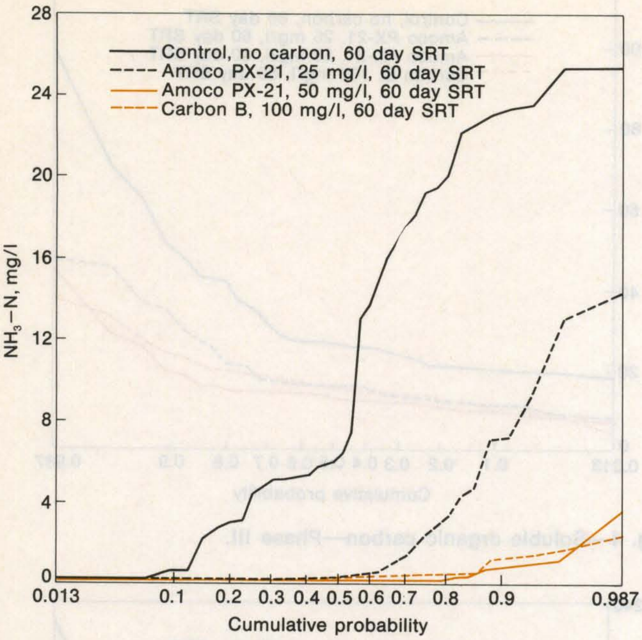

Fig. 6—Ammonia-Nitrogen—Phase III.

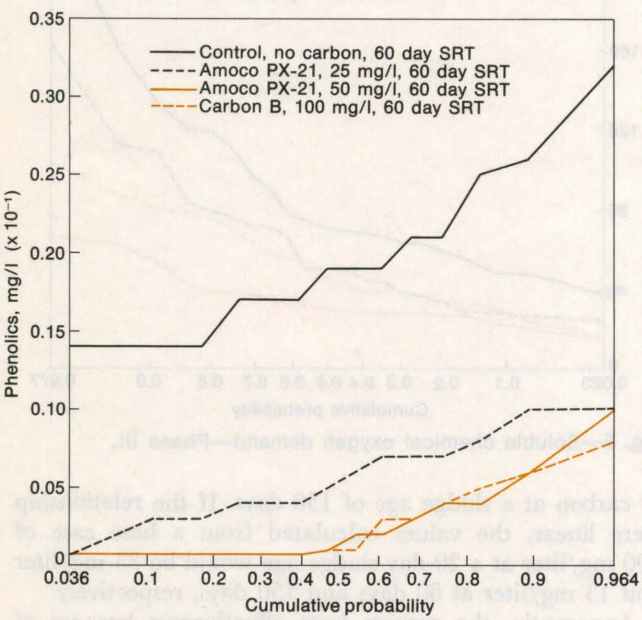

Fig. 7—Phenolics—Phase III.

the 98% level. For BATEA the daily maximum variability factors for TOC, COD, NH_3-N, and phenolics are proposed at 1.6, 2.0, 2.0, and 2.4, respectively. The 30-day maximum values are 1.3, 1.6, 1.5, and 1.7, respectively.

TABLE 8—Phase III—BATEA guideline and actual variability factors for pilot plant fed 25 mg/liter of carbon A1

Parameter	BAT Guideline Variability Factor		Actual Variability Factor	
	Daily Max.	30 Day Max.	Daily Max.	30 Day Max.
Soluble Organic Carbon......	1.6	1.3	2.8	2.8
Soluble COD........	2.0	1.6	7.5	7.5
NH_3-N........	2.0	1.5	2.1	2.0
Phenolics........	2.4	1.7	5.0	5.0

Figs. 4, 5, 6 and 7 show probability data for the 30-day operating periods during Phase III. Table 8 shows the daily maximum (99% probability) and 30-day maximum (98% probability) variability factors calculated from these figures for the plant fed with 25 mg/liter of Carbon A1. The EPA guideline values are also given. The actual variability factor was calculated as the 99% (or 98%) probability value divided by the target quality value. In general, the variability in effluent quality was higher than the guideline values.

It is important to note that the proposed guideline variability factors are unrealistic. The data base used by EPA[1] for their production was obtained from limited pilot studies. In addition, BPTCA 30-day maximum (98% probability) values were used as the BATEA 30-day maximum values. Variability factors will undoubtedly have to be amended before BATEA *goals* become BATEA *standards.*

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