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Odor Control Performance of Carbon-Loaded Rayon Monofilaments

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The preparation of activated carbon-loaded rayon monofilaments for use as adsorbing filters in air purification has been described by McDowell.[1] In the evaluation of the efficacy of such devices, it is necessary to consider the properties of the filter, the engineering practice in air handling, and the demands of the environment. When the filter is used to protect an indoor space from outdoor odorous vapors, its one-pass efficiency must be sufficiently great to reduce the odor to or below the threshold concentration, as determined by the relationship:

$$E = [(C_i - C)/C_i] (100) \quad (1)$$

where E is adsorption efficiency (per cent), C_i is imput concentration, and C is concentration of odorant in filtered air stream.

As an example, assume an outdoor space in the vicinity of a gas odorizing site is contaminated with ethyl mercaptan to a concentration of 0.1 ppm. Assuming a value[2] for threshold concentration of $0.26(10)^{-3}$, the required efficiency for a carbon filter to protect the occupants of a building from odor by purifying the intake air will be

$$E = \frac{0.1 - 0.26(10)^{-3}}{0.1} (100) = 99.8\%$$

If the odorant were cresol (threshold 0.72×10^{-2} ppm),[3] the required efficiency would be 92.9%. It is thus clear that high orders of efficiency may be needed for protection of spaces from outdoor odors.

When odors are generated in an occupied space (tobacco, food, body, cosmetics, materials of construction), and air is handled by recirculation through a carbon filter, the situation is quite different. In such case, removal of odor by adsorption and formation of odor by generation proceed simultaneously, and, if the rates remain constant, an equilibrium is set up. If the equilibrium concentration is less than the odor threshold concentration, the space is said to be deodorized. The equilibrium relationship[3] between generation and adsorption rates and filter efficiency in a closed space is given by

$$C_\infty = G/EQ \quad (2)$$

where, C_∞ is odorant concentration at equilibrium, Q is the volumetric rate of air flow through adsorber, and G is the time rate of generation of odorants. Only if C_∞ is equal to or less than the odor threshold concentration will the space eventually be deodorized. In practice, a light-duty adsorber will never deodorize a room in which generation rate is high (e.g., animal laboratory), but will be more or less effective in ordinary occupied spaces (residences, offices, conference rooms, etc.). Other things being equal, a more efficient filter will be able to cope with a higher rate of odor generation and will approach equilibrium at a lower odorant concentration.

The relationship expressing the concentration at any finite time for a specific initial concentration of odorant is:

$$C = C_0 e^{-EQt/V} + (G/EQ)(1 - e^{-EQt/V}) \quad (3)$$

where C is the concentration at any time, C_0 is initial concentration, t is time, and V is the volume of space. If no odor is generated in the space ($G = 0$), then the concentration will approach zero according to the equation:

$$C = C_0 e^{-EQt/V} \quad (4)$$

Therefore, the more efficient the filter, the lower will be the odorant concentration at any finite time, whether or not odor is generated continuously in the space.

The capacity of an adsorber may be taken as the total quantity (weight) of odorant it will retain while giving satisfactory performance or the duration of service before it must be replaced or reactivated. The latter information is of more practical importance, but requires a knowledge of prevailing vapor concentrations:

$$t = SW/EQC \quad (5)$$

where t is the service life of filter, S is the ultimate proportionate saturation of carbon with odorant or retentivity (fractional), and W is weight of adsorbent. Filter efficiency thus plays an important role in determining both performance and life.

Adsorbent filters designed for air purification in occupied spaces are expected to reduce vapor concentrations from spaces in which initial concentrations are often too low for any direct chemical or physical measurement. If, for testing purposes, vapor concentrations are raised to comparatively high levels, then any appraisal of performance will be subject to criticism of invalidity because of an unrealistic environment. In this study, it was decided to use low vapor concentrations, approaching those which might exist in an odorous occupied space, and to make sensory odor judgments, in a dynamic system of known initial concentration, as functions of adsorbent bed depth and air detention time within the filter bed. This scheme would bypass any problems caused by the use of unrealistically high vapor concentrations. The results of such sensory tests can also lead to estimates of adsorption efficiencies, on the following basis.

Assume, as a working hypothesis, the validity of the Weber-Fechner law of perception, which, applied to odor, states that olfactory intensity is proportional to the logarithm of the stimulus (odorant concentration). Then,

$$\log C = (1/K)I + b \tag{6}$$

where I is the perceived odor intensity, $1/K$ is the slope of the plot of log C versus I, b is the value of log C when I is zero, or log C_t, and C_t is the odor threshold concentration. Then,

$$\log C = (1/K)I + \log C_t$$

and

$$I = K \log C/C_t \tag{7}$$

If the value of C_t is measured (here I is zero, by definition), and the highest value of C is also measured (I will have its corresponding highest value), then it will be possible to determine any C/C_t value from intensity measurements made on a "linear" perceptual scale.

For any measurement,

$$C = (C/C_t)\, C_t$$

The dilution ratio C_i/C_t can be measured; let it be designated D. Then

$$C_i = DC_t$$

and

$$E = 100\ (D\text{-}C/C_t)/D \tag{8}$$

The input concentration is, of course, the highest concentration, and can be determined by the method of odorant injection rather than by chemical analysis. Since any other concentration may be determined from the C/C_t value, it should be possible to estimate adsorption efficiency without chemical analysis.

If the adsorber can be divided into linear increments of bed depth and odor intensity determinations be made after each increment, it should be possible to show the pattern of odorant reduction through the adsorbent bed. This will also give the critical bed depth (minimum depth needed for deodorization) under given flow conditions. Finally, the time movement of this pattern is the adsorption wave,[4] which gives a realistic picture of filter life or capacity. Since the drift will ordinarily be slow, it is experimentally convenient to accelerate it. Such acceleration is permissible so long as it does not occur during the time when odor intensity judgments are being made.

Experimental Results

Odorant. Tobacco odors are generally considered to be the most significant ones in occupied spaces. A "real" tobacco odor made up, for example, of a mixture of stale cigar and cigarette butts, would not be reliably reproducible. Instead, a simulated smoking room malodor[5] of known composition was used (Table I).

TABLE I
Composition of Simulated Smoking Room Malodor

Component	Parts by weight, %
Propylene glycol (odorless)	49.0
Furfural	4.9
Furfuryl mercaptan	0.1
Methylethylpyridine	12.3
Thiovanic acid	0.5
Creosote	3.7
Pyroligneous acid	29.4
	99.9

Adsorbent Filters. Four types of filter media were used (see Table II): rayon filament loaded with activated carbon of the type used for (*a*) air purification (Sample 1) or (*b*) water purification (Samples 2 to 5); (*c*) granular 6–14 mesh air-purification activated carbon (Sample 6); and (*d*) sec-

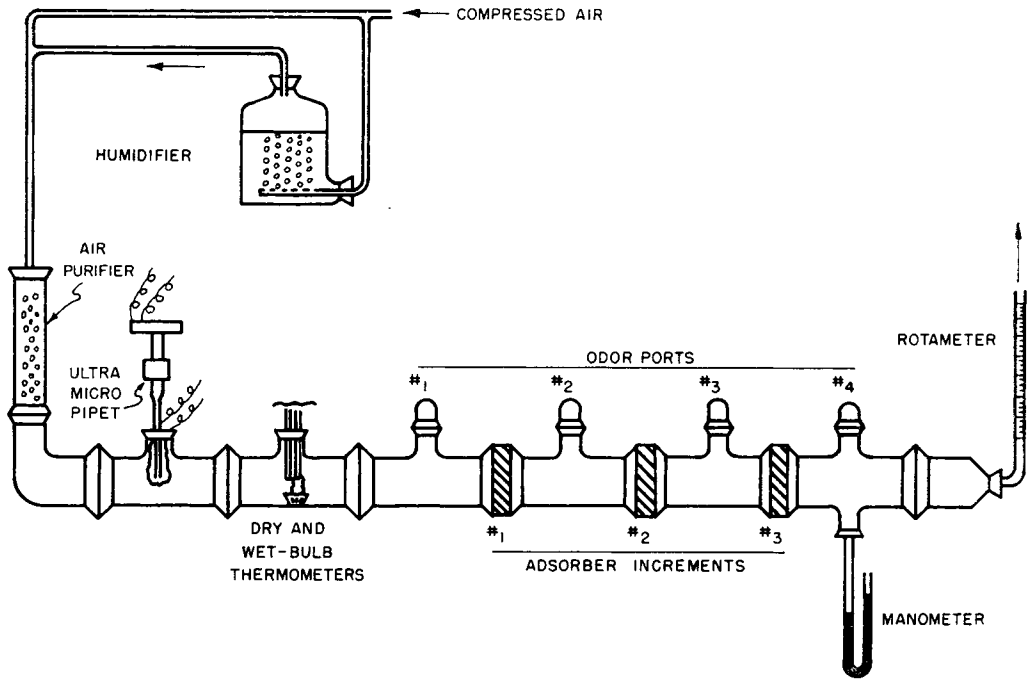

Fig. 1. Incremented carbon bed apparatus.

tions taken from filters manufactured for use with unit air conditioners, comprising an open lattice of support material impregnated with carbon (Samples 7 and 8).

TABLE II
Adsorbent Filter Compositions

Sample	Composition wt.-%	
	Support medium	Carbon
1	Rayon, 17.6	82.4
2	Rayon, 26.2	73.8
3	Rayon, 20.7	79.3
4	Rayon, 22.4	77.6
5	Rayon, 21.1	78.9
6	None	100.
7	Paper, ~ 75	~25
8	Not determined	Not determined

The carbon-loaded rayon filters were packed to a uniform density of 0.096 g./ml. by the following procedure. The filter material was dried overnight in a vacuum desiccator and the calculated weight (8.25 g. for a $^3/_4$-in. bed of 3-in. diameter) was wetted with sufficient water to plasticize it, packed uniformly into a borosilicate glass $^3/_4$ in. × 3 in. cylinder (see below), and retained therein by disks of aluminum screening. The packed spacer was dried in an oven at about 100°C., allowed to cool to ambient temperature, and then used directly in the testing apparatus (if dry) or equilibrated with moisture in an atmosphere whose relative humidity corresponded to that of the gas stream to be tested.

Equipment. The increments of adsorbent bed, the system for odorant injection into the air stream, the ports for sampling the stream for odor judgments, and miscellaneous auxiliary equipment

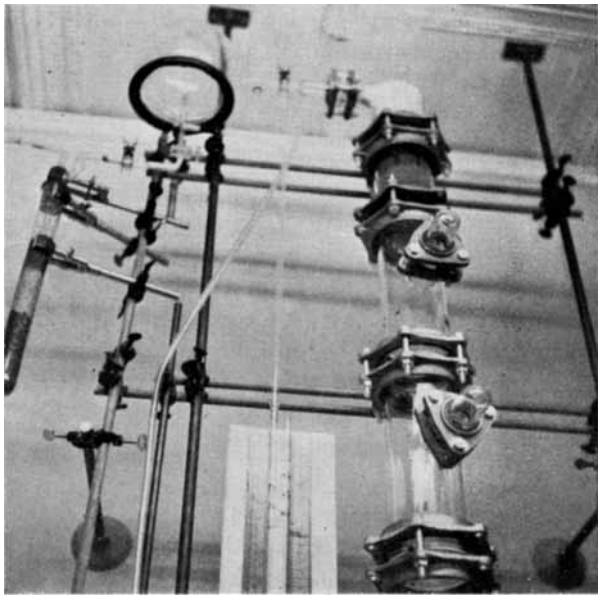

Fig. 2. Photograph of equipment.

were all incorporated in a borosilicate glass pipe system of 3 in. internal diameter (Figs. 1 and 2). Compressed air (Fig. 1) was humidified, if necessary, by metering a portion of it through water until the desired humidity was obtained, and was then purified. For dry air, the purifier was packed with (in order of contact with the air stream) indicating silica gel, granular activated carbon, and glass wool. For humid air, the silica gel was omitted, and the carbon was previously equilibrated with an atmosphere of the same aqueous tension as that to be reached in the experiment. Odorant was introduced into the air stream by means of an ultramicropipet (Emil Greiner Co., New York) driven by a synchronous clock motor at 1 rpm or 1 rph. Screens were inserted at the two flange connections which followed the odorant injection to help achieve turbulent flow. The three increments of adsorbent bed were contained in $^3/_4$ in. glass pipe spacers. Ports for odor sampling were available before and after each bed increment.

At a motor speed of 1 rph, normal evaporation kept pace with odorant delivery; at motor speed of 1 rpm it was necessary to aid evaporation by impinging the delivered odorant onto a warmed surface of nichrome ribbon. Concentration of odorant in the gas stream going into the carbon bed is given by:

$$C_i = 22.4 Q_i d C_L / M Q_t \qquad (9)$$

where C_i is input concentration (fractional, multiply by 10^6 for ppm), Q_i is delivery rate of liquid odorant, d is the density of liquid odorant, C_L is the concentration of liquid odorant in excess diluent (fractional), M is the average molecular weight of odorant, and Q_t is the rate of flow of the air stream.

The various factors may be evaluated as follows. Delivery rate of liquid odorant, Q_i, is fixed by the capacity of the pipet (0.1 ml.), the total number of revolutions (32), and the motor speed. Then, at 1 rpm, Q_i = 0.0031 ml./min., and at 1 rph, Q_i = 0.000052 ml./min. Density, d, of liquid odorant is 1.0 g./ml. Average molecular weight of the liquid odorant, estimated by taking a weighted average of its component molecular weights, is 80.

Total flow rate, as measured by the rotameter, was maintained at 53.3 l./min.

In all runs except those which were used for determination of odor threshold concentration, the odorant was used in the composition specified by Table I without further dilution; hence $C_L = 1$. For the threshold measurements, the odorless component of the mixture (propylene glycol) was used as the diluent.

Substituting these values for the constants in eq. (9), we have:

At 1 rpm:

$$C_i = \frac{22.4 \times 0.0031 \times 1 \times 10^6}{80 \times 53.3}$$

$$= 16.3 \text{ ppm}$$

At 1 rph:

$$C_i = 0.27 \text{ ppm}$$

Flow Rate and Detention Times. The linear flow rate is the volumetric flow rate divided by the cross-sectional area, or

$$\text{Linear flow rate} = \frac{53.3 \text{ l./min.}}{28.3 \text{ l./ft.}^3 \times 0.049 \text{ ft.}^2}$$

$$= 38.6 \text{ ft. /min.}$$

Bed porosity, or per cent voids, must be known to calculate detention times. The porosity p is given by

$$p = \frac{\text{total volume} - \text{adsorbent volume}}{\text{total volume}}$$

$$= (87.0 \text{ cm.}^3 - 2.29 \text{ cm.}^3)/87.0 \text{ cm.}^3$$
$$= 0.975$$

Air detention time, t_d, in seconds, is given by:[6]

$$t_d = 60 Apl/Q \qquad (10)$$

Here l is bed thickness (one spacer is 1.9 cm.), A is bed area, (46 cm.^{2}), Q is air flow rate, bed porosity p = 0.975, and

$$t_d = \frac{60 \text{ sec./min.} \times 1.9 \text{ cm.} \times 46 \text{ cm.}^2 \times 0.975}{53.3 \text{ l./min.} \times 1000 \text{ cm.}^3/\text{l.}}$$

$$= 0.096 \text{ sec. per increment of bed.}$$

Odor Intensity Reference Scale and Judgment Procedure. To anchor the intensity ratings to a scale of materially fixed concentrations,[7] a series of logarithmically increasing dilutions of the tobacco odorant was made according to the following schedule:

Bottle No. 1: 1 ml. "tobacco"
Bottle No. 2: 1 ml. "tobacco" + 1 ml. propylene glycol
Bottle No. 3: 1 ml. from bottle No. 2 + 1 ml. glycol
Bottle No. 4: 1 ml. from bottle No. 3 + 1 ml. glycol
Series is continued to bottle No. 15.

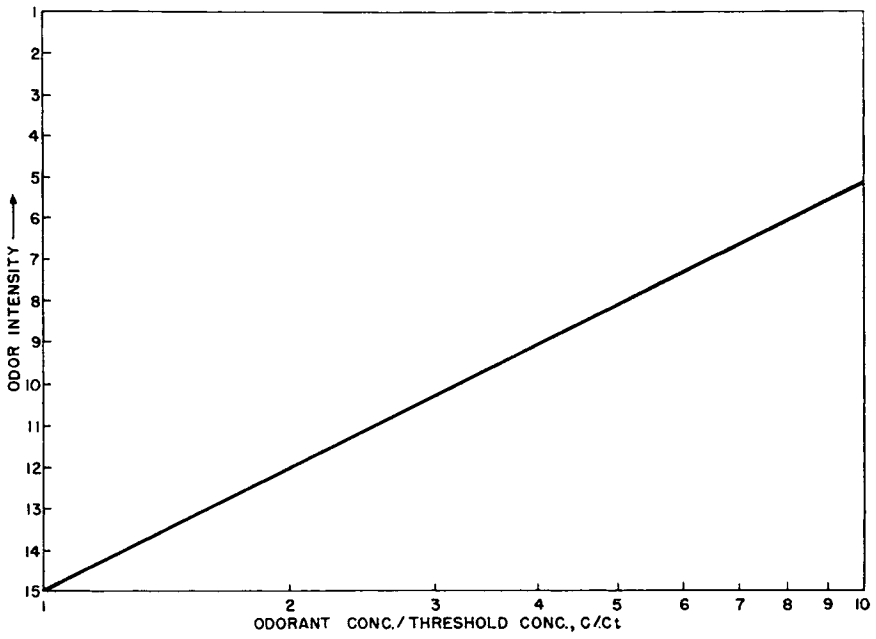

Fig. 3. Odor intensity vs. C/C_t.

In terms of the original odorant, the concentration of any solution is $2^{(1-n)}$, where n is the bottle number in the series given above. The lower numbers, of course, correspond to higher odor intensities.

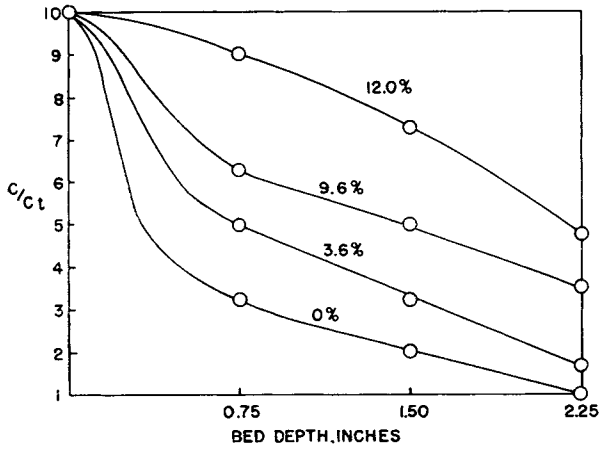

Fig. 4. Deodorizing performance, sample 1, dry air, 90°F.

To judge the odor intensity at a particular part in the air filter system, the port cap is removed, and the judge is asked to select the bottle whose intensity best matches that of the air escaping from the port. The number on the selected bottle is taken as the odor intensity score. Half-numbers may be used for intermediate scores. Each judge works independently and enters his score without knowledge of the scores obtained by others.

Threshold Concentration of Tobacco Odorant. The threshold concentration was determined by selecting the highest bottle number (lowest concentration) from the intensity scale which, when

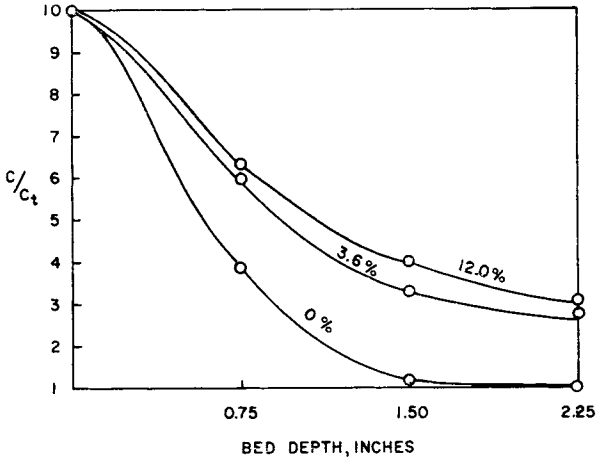

Fig. 5. Deodorizing performance, sample 1, 80% R.H., 80°F.

inserted into the air stream from the microburet at the lowest delivery rate, would give a recognizable odor. Final adjustment was made by regulating total air flow until threshold was reached. Necessary total flow was 68.2 l./min. The solution used was taken from bottle No. 4. Hence, concentration = 2^{-3}, or 0.125, and, from eq. (9),

$$C_i = \frac{22.4 \times 5.2 \times 10^{-5} \times 1 \times 0.125 \times 10^6}{80 \times 68.2}$$

$$= 0.027 \text{ ppm}$$

and, at 1 rph delivery, $C_i/C_t = D = 10$.

The minimum detectable concentration in the reference odor series was bottle No. 15. The full-strength odorant, injected into the system at 1 rph, gives an intensity from the first sampling port which corresponds to bottle No. 5. These two points fix the reference line shown in Figure 3.

Figures 4 to 10 present performance data for activated carbon samples under the conditions specified. Each point represents the C/C_t value corresponding (from Fig. 3) to the mean odor intensity score among a panel of 4 to 5 judges. The per cent figure for each curve denotes the degree of saturation (based on the first bed increment) of the adsorbing medium with the tobacco malodor.

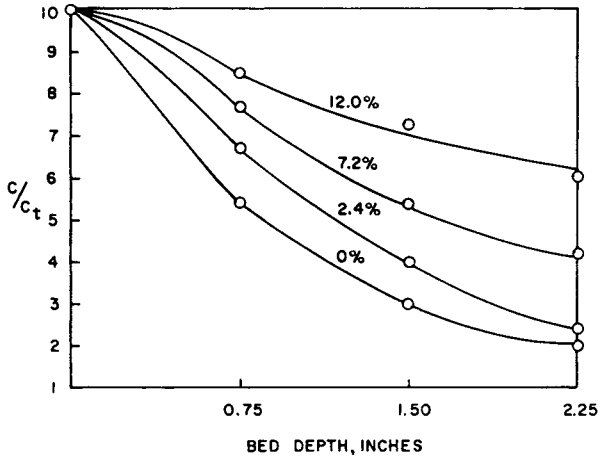

Fig. 6. Deodorizing performance, sample 2, dry air, 90°F.

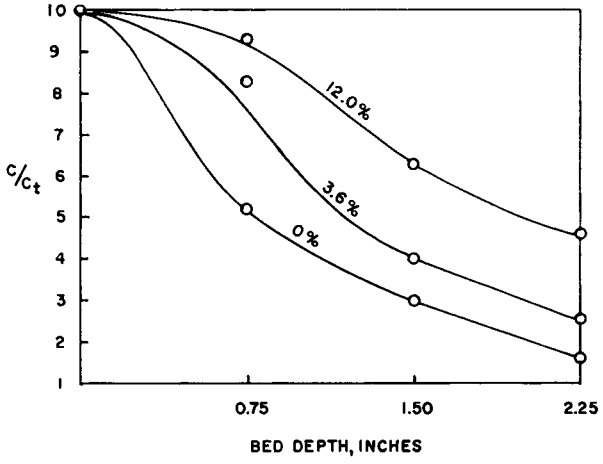

Fig. 7. Deodorizing performance, sample 3, dry air, 90°F.

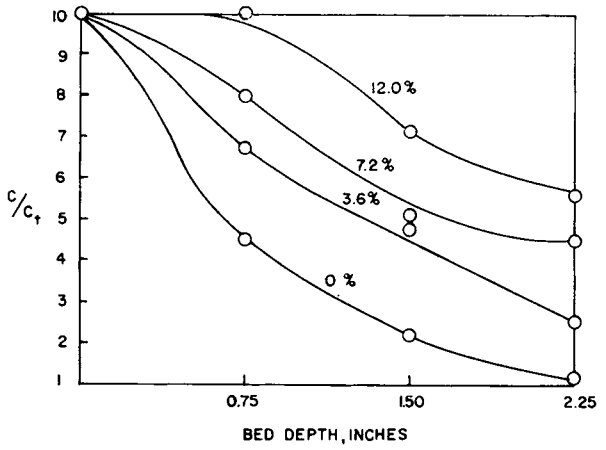

Fig. 8. Deodorizing performance, sample 4, dry air, 90°F.

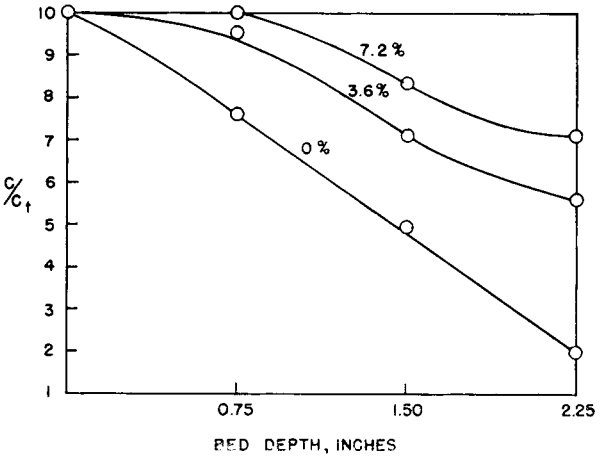

Fig. 9. Deodorizing performance, sample 5, dry air, 90°F.

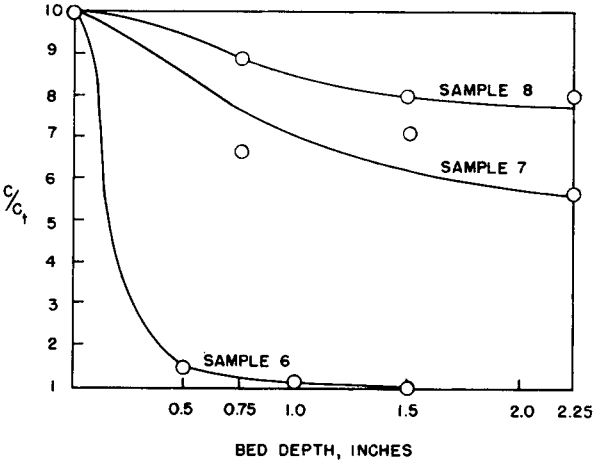

Fig. 10. Deodorizing performance, samples 6, 7, and 8, dry air, 80°F.

The figure "0%" therefore represents initial performance of the fresh filter.

These plots are thus experimentally determined adsorption waves, showing performances of the filter beds with advancing degrees of saturation. Each set is linearly adjusted, when necessary, so that the intensity at port No. 1 is taken as 5.0; the intensity increments between ports remain unchanged.

Conclusions

Reliability of Data. Each judge was able to distinguish between adjacent points on the intensity scale 100% of the time, except at or within two points of the odor threshold concentration. This ability could be tested by removing one bottle from the intensity series and presenting it to the blindfolded judge. He was then given the task of replacing the bottle in its correct position in the concentration series, according to odor intensity. After training, this task could always be accomplished successfully.

During the experimental program, test results were entered independently by each judge. To eliminate transfer of information by reduced visible cues, some of the tests were carried out by allowing only the individual making the judgment to occupy the laboratory. In other tests, each judge was blindfolded and was placed at each of the four odor ports in random and unrevealed (to him) order. It was found that the scatter of the data was not materially changed by these procedures, but that the experimental program was significantly retarded; these extreme precautions were therefore abandoned.

In general, the range of intensity judgments by a panel of 3 to 5 testers was between 0.5 and 1.5. This is much more precise than the usual "subjective" odor intensity data which uses a scale anchored in word definitions alone.

Since the intensity data reported are the averaged jury scores, it is important to consider the reliability of the differences between any two averages. A typical case involving a three-membered panel is taken from the raw data from Figure 6 at 4.8% saturation: Analysis by the t-test shows that the difference between the mean intensity ratings of ports 1 and 2 is very highly significant, much above the 99% level; the difference between ports 2 and 3 is highly significant, also above the 99% level; between ports 3 and 4 the difference is significant at the 90% confidence level. In general, typical data show significant differences between intensity means which differ by one unit or more.

Comparative Performance of Filters. A good adsorbing filter should perform efficiently and have a high capacity. In terms of the measurements herein described, this means that the adsorption wave should dip sharply with bed depth, and should progress only slowly to the right with increasing carbon saturation. Next to the granular carbon, the most effective adsorbent was Sample 1, the rayon loaded with air-purification carbon, which performed well under both dry and humid conditions. The superior performance under humid conditions may be related to the dissipation of heat of adsorption by the desorption of some of the water.[8] The filters comprising rayon loaded with water-purification carbon (Samples 2 to 5) showed a range of performance qualities, but as a class were poorer than Sample 1. All of the carbon-loaded rayon compositions, however, were distinctly superior to the two open-lattice filters, whose initial performance is shown in Figure 10.

Estimation of Filter Efficiency. From eq. (8), $D = 10$ and

$$E = 100 - 10C/C_t \qquad (11)$$

Thus, for example, the adsorption efficiency in the first passage through the first increment of the Sample 1 bed, under dry conditions (Fig. 4) is

$$E = 100 - 10\ (3.2) = 68\%$$

The efficiencies of the commercial open-lattice filters, Samples 7 and 8 in Figure 10, and 26% and 11%, respectively.

Projection to Service Performance. Filter performance in service depends on the properties of the environment as well as on those of the filter, as outlined in the introductory paragraphs. In air recirculating systems, a high filter efficiency means a rapid odor reduction. Under extreme conditions of very high filter efficiency and rapid air change, spectacular performance can indeed be achieved.[9] Other things being equal, therefore, it is important to attain as high a filter efficiency as possible.

The data presented here show that, for approximately the same carbon loading, activated carbon which has properties designed for use in air purification transfers these attributes to the rayon filament in which it is incorporated, and is therefore preferable to a water-purification carbon for use in air systems.

For a given variety of activated carbon, the total capacity of the loaded rayon will depend on the

total quantity of carbon. Hence, competitive carbons may be rated on a cost per unit weight of odorant adsorbed basis. The property of practical interest, however, is service life, which involves more than merely capacity to adsorb. Service life may better be considered to be the duration of acceptable service offered by a given filter in a given environment. According to this concept, a filter whose efficiency is poor, initially or after a short time, has a low service life even though its total capacity (on a weight basis) is high. It is therefore important, in the design of adsorbing filters for use in human-occupancy systems, that carbon which provides the most efficient overall performance be used.

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Synopsis

The odor control performance of carbon-loaded rayon monofilaments depends on the type and proportion of activated carbon used. The filter efficiency in turn determines the rate of odor reduction in a closed space and the ultimate or equilibrium concentration when odorant generation and removal occur simultaneously. Four types of filters were evaluated: rayon loaded with air- or water-purification carbon, granular air-purification carbon, and open-lattice commercial filters. Air laden with synthetic tobacco malodor was streamed through successive increments of adsorbent bed. Sensory odor judgments were made before and after each adsorption stage by matching the effluent odors with those from mixtures of known concentrations. It was thus possible to estimate adsorption efficiencies and, by repeating the measurements after various degrees of carbon saturation, to construct adsorption waves. It was found that air-purification carbon was superior to water-purification carbon for loading in the rayon filaments, and that all the rayon-carbon compositions were much superior to the commercial open-lattice filters. The rayon-carbon filters showed efficiencies up to 70% for one pass at 0.1 sec. detention time through a $^3/_4$-in. bed packed at 0.096 g./ml. with a filament comprising 20% rayon and 80% carbon.

Résumé

L'aptitude pour des monofilaments de rayonne chargée de carbone à controler les odeurs, dépend du type et de la proportion de carbone actif employé. L'efficacité du filtre détermine la vitesse de suppression de l'odeur dans un espace clos, et détermine la concentration limite ou concentration d'équilibre lorsque la formation et la disparition de l'odeur ont lieu simultanément. Quatre types de filtres sont étudiés: la rayonne traitée au carbone (*a*) à purification par air (*b*) ou par eau, (*c*) au carbone en granule à purification par air et (*d*) les filtres commerciaux à réseaux libres. On envoie de l'air chargé d'odeur de tabac à travers des couches successives d'absorbants. Des estimations sont faites avant et après chaque étape d'absorption en comparant les odeurs effluentes avec celles provenants de mélanges de concentrations connues. Il est donc possible d'estimer les efficacités d'absorption et, en recommençant les mesures après divers degrés de saturation en carbone, de construire des courbes d'adsorption. On trouve que le carbone à purification par air est supérieur au carbone à purification par eau pour traiter les filaments de rayonne, et que toutes les compositions rayonne-carbone sont de loin supérieures aux filtres commerciaux à réseaux libres. Les filtres rayonne-carbone ont une efficacité supérieure à 70%, pour un passage, pour un temps de rétention de 0.1 seconde à travers un lit de $^3/_4$ pouce formé à 0.096 g/ml de filament à 20% de rayonne et 80% de carbone.

Zusammenfassung

Die Wirksamkeit der Geruchskontrolle durch kohlebeladene Rayon-Einzelfasern hängt von der Art und Menge der verwendeten Aktivkohle ab. Die Filterwirksamkeit bestimmt ihrerseits die Geschwindigkeit der Geruchsverminderung in einem geschlossenen Raum, sowie die Rest- und Gleichgewichtskonzentration, wenn die Bildung und Entfernung des Geruchsträgers gleichzeitig verlaufen. Vier Filtertypen wurden untersucht: Rayon beladen mit (*a*) Luft- oder (*b*) Wasserreinigungskohle, (*c*) körnige Luftreinigungskohle und (*d*) offene käufliche Filter. Mit synthetischem, üblen Tabakgeruch beladene Luft wurde durch sukzessive vergrösserte Adsorptionsschichten geschickt. Riechproben wurden vor und nach jeder Adsorptionsstufe durch Vergleich des ausströmenden Geruches mit dem von Mischungen bekannter Konzentration gemacht. So war es möglich, die Adsorptionswirksamkeit zu bestimmen und durch wiederholte Messungen bei verschiedenem Sättigungsgrad der Kohle Adsorptionskurven aufzustellen. Es wurde gefunden, dass Luftreinigungskohle der Wasserreinigungskohle bei der Beladung von Rayonfasern überlegen war und dass alle Rayon-Kohle-Systeme viel besser waren als die käuflichen offenen Filter. Die Rayon-Kohle-Filter zeigten Aufnahmen bis zu 70% bei einmaligem Durchgang und einer Kontaktzeit von 0,1 Sekunden in einer $^3/_4$″ dicken Schicht, gepackt bei 0,096 g/ml mit einer Faser, die aus 20% Rayon und 80% Kohle besteht.