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Part II: Steam-Carbon Reaction Kinetics

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The kinetics of the reaction between steam and spent activated carbon (from sucrose deposition) was studied at 1003 to 1123 K and 1 atm pressure. At these conditions some virgin carbon, upon which the sucrose was originally adsorbed, was also gasified. Rates of oxidation were essentially the same for spent and virgin activated carbon.

The kinetics data fit a Langmuir-Hinshelwood equation, originally proposed for gasification of other types of carbon, with an activation energy of 2.3×10^5 J/mol.

Surface area and pore volume measurements indicated that the adsorption capacity of the original carbon could be completely restored by thermal regeneration followed by reaction with steam. However, some activated carbon is lost implying that there would be an optimum extent of steam gasification in a cyclical adsorption-regeneration process.

SCOPE

Kinetics studies are reported in Part II for the reaction between steam and carbon remaining after spent activated carbon has been thermally regenerated. The samples used were those dried by method B (drying at 393-403 K for three days, see Part I) and then thermally regenerated with helium in the TGA equipment at a rate of temperature increase of 0.3 K/s. When the temperature obtained a desired level (between 1003 and 1123 K) the rate of the steam carbon reaction was evaluated by maintaining the samples in the TGA apparatus and measuring their weight change at constant temperature. During this reaction period a gas mixture of constant composition, consisting of helium, steam and hydrogen (for some runs) was passed over the sample. Rates of reaction were determined for steam concentrations from 0.04 to 6.0 mol% and hydrogen concentrations from 0 to 0.5 mol%. As indicated in Figure 6 of Part I, there was essentially no thermal decomposition between 773 and 1123 K. Hence, the runs made for the steam-carbon reaction over this temperature range were carried out with carbon samples of the same characteristics, even though they had been subjected to different temperatures of thermal decomposition.

The kinetics of the noncatalytic, steam-carbon reaction have been studied with many types of carbon (activated carbon from coconut shell charcoal—Gadsby et al., 1946 and Long and Sykes, 1948, 1950; coke from coal—Hunt et al., 1953; graphite from electrodes—Binford and Eyring, 1956). Interest in coal gasification has been the impetus for numerous investigations of both the H_2O-C and CO_2-C reactions; for example, Gadsby et al. (1948), Lewis et al. (1949), Goring et al. (1952), Johnstone et al. (1952), Pilcher et al. (1955), and Ergun (1956). The need to retard gas-carbon reactions in atomic reactors has stimulated numerous other kinetics works including those of Overholser and Blakely (1965), Blakely and Overholser (1965), Malinauskas (1970), and Strange and Walker (1976). Finally, the recent interest in regeneration of activated carbon used for water purification is leading to kinetics studies of the reaction between steam and thermally regenerated activated carbon (for example, Klei et al., 1975). As noted in Part I, thermal regeneration to 773 K reduces the adsorbed organic compounds to residual carbon. Hence, in the subsequent reaction with steam both residual carbon and base carbon can react. It is of interest to compare the rates of reaction for these two kinds of carbon.

Rate data for the reaction of carbon, from many sources, with steam follows the equation

$$R = \frac{k_1 p_{H_2O}}{1 + k_2 p_{H_2} + k_3 p_{H_2O}} \qquad (1)$$

which has been proposed by Walker et al. (1959) and by Ergun (1962, 1965). However, there are large variations in the magnitude of the rates, the activation energy for k_1 and the effect of temperature on k_2 and k_3. Also, there is disagreement on the mechanism of the elementary processes leading to Eq. 1. It is generally accepted that carbon monoxide is produced at the carbon surface, but part of this CO may be converted to carbon dioxide by the water gas-shift reaction.

CONCLUSIONS AND SIGNIFICANCE

Our rate data for the regeneration of *activated* carbon with steam could be correlated with a Langmuir-Hinshelwood equation previously proposed for the steam-carbon reaction. The *measured* rate was the same for virgin activated carbon (coal-based BPL carbon from Calgon Corporation) and for thermally regenerated particles containing residual carbon from sucrose adsorption. Over the mass range of two to three times the amount of residual carbon, the mass decreases linearly with time.

The rate changed from first order to zero order with respect to steam as the water vapor concentration was increased from 0.04 to 6.0%. Hydrogen retarded the rate. Both effects are predicted by the Langmuir-Hinshelwood equation. The temperature dependency of the parameters in this rate equation indicated an activation energy of 2.3×10^5 J/mol.

By following thermal regeneration with reaction with steam, surface areas greater than that of the original virgin carbon

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could be attained. However, even for regeneration to the same surface as for virgin carbon there was some gasification of virgin carbon.

The results of Parts I and II establish rate equations for complete thermal and steam regeneration of BPL activated carbon loaded with sucrose.

The measurements in Part II were of two types: TGA experiments at constant temperature, as mentioned in the SCOPE, and surface-area and pore-volume data to evaluate the extent of regeneration.

EXPERIMENTAL

TGA Experiments

Samples (virgin carbon and B samples) were thermally regenerated to the reaction temperature (1003 to 1123 K) in the TGA apparatus shown in Figures 1a and 1b (Part I). When the desired temperature was reached, the gas stream was changed from helium to a helium-steam mixture to which hydrogen was sometimes added. Then the temperature was held constant and the sample weight measured continuously. The steam-helium mixture was prepared by diverting part of the helium flow through a saturator containing liquid water at a constant temperature. The stability and absolute value of the water-vapor concentration were checked with a gas chromatograph. The flow rate of each stream was 6.7×10^{-7} m^3/s (at 298K and 1 atm).

In these experiments gases of two different compositions entered the TGA through the two feed positions (Figure 1b). This introduced the possibility of incomplete mixing of the streams before they reached the sample, and also the possibility of concentration gradients in the gas phase around the carbon particles. The second possibility was tested by making preliminary runs with a different number of layers of carbon particles in the sample holder. These runs were made without the flange (Figure 1b) used later to prevent incomplete mixing of the feed streams. Rate data from these experiments with a different number of layers are shown as solid points on the left side of Figure 1. There is a 40% decrease in rate when 6 layers are used rather than a single layer. Hence, there is a retardation of the rate due to mass transfer when multiple layers are used. The final kinetics data were obtained with one layer of particles in the holder.

The data points on the right side of Figure 1 show the effect of mixing of the two streams. The upper, solid points [runs 5-S and 6-S] represent data when pure helium was fed through the balance and 0.3% H_2O in helium was fed through the side arm (figure 1b) with no flange. Run 16-S is for the same flow arrangement but with the flange. The higher rate for runs 5-S and 6-S suggests that, in the absence of the flange, steam reaches the sample without complete mixing. Run 18-S was made with the flange but with the flow streams combined upstream. Since the results for runs 18-S and 16-S agree reasonably well, it was concluded that the flange was sufficient to give good mixing of the feed streams. Also, the activation energy obtained from the data agreed with published values. If mass transfer influenced the data, the activation energy should have been lower. In all the runs the total, combined flow rate was the same.

Small amounts of oxygen in the gas streams can distort the results for the carbon-steam reaction, because the carbon-oxygen reaction is relatively fast. Hence, attempts were made to reduce oxygen contamination. An ultra high-purity helium (99.999% purity) was employed for Part II experiments. The oxygen content is stated to be less than 1 ppm. Hydrogen purity was 99.95%, but since its maximum concentration was 0.5%, oxygen contamination from this source was also low. The major source of oxygen appears to be from leaks of air into the system. An estimate of O_2 content from gas-chromatographic analysis gave a value of 5-10 ppm. Some weight loss of carbon sample was observed when the TGA apparatus was operated from 1003 to 1123 K and only helium was in the gas feed. Assuming this loss in weight is due to oxygen, a similar concentration of 5 to 10 ppm is indicated. The rates for the steam-carbon reaction were corrected for this effect, which usually amounted to less than 30% (Table 1). The rates are based upon the mass of virgin carbon remaining unburned when the samples reach reaction temperature.

Surface Area and Pore Volume Data

To measure the effectiveness of the steam-carbon reaction for regeneration, surface areas and pore volumes were measured for samples exposed to various reaction conditions. A Sorptometer was used for both determinations. Surface areas were established by the single-point BET method described in Part I. Pore volumes were determined by measuring the amount of nitrogen adsorbed (at liquid N_2 temperature) at a relative pressure, with respect to the vapor pressure, of nitrogen equal to 0.49. At this condition, the amount adsorbed was taken as that equal to surface adsorption and to condensation in pores less than a radius of 2.2 nanometers, as suggested by Dollimore and Heal (1964).

GASIFICATION RATES FOR SPENT AND VIRGIN CARBON

Figure 2 shows illustrative weight vs. time data for virgin carbon (Runs 13-V and 8-V) and spent carbon from B Samples (Runs 11-S and 7-S). These results were obtained by first heating the samples to 1002 K in the TGA apparatus at a rate of 0.3 K/s with a flow of helium. The weight loss between 773 and 1002 K is due to vaporization or desorption of extraneous substances, as explained in Part I. After this preheating step, for the curves labeled B-He and V-He, the flow of pure helium was continued while the temperature was maintained constant at 1002 K. The curves labeled B-H_2O and V-H_2O were obtained by changing the gas stream from helium to a 0.3% H_2O and 99.7% He mixture when the temperature reached 1002 K, and then operating at the constant level of 1002 K.

The difference in weight at $t = 0$ between the virgin carbon and B samples represents the residual carbon after thermal regeneration of the B samples. This amounts to about 1.5% of the initial mass of the virgin carbon used in preparation of the samples. The decrease in mass shown by the straight line sections (at large t values) of the curves B-He and V-He is due to the

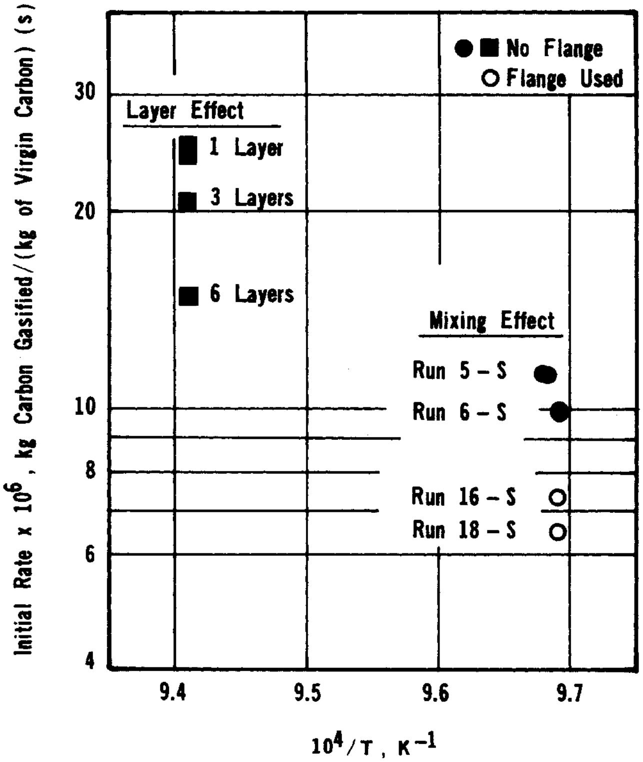

Figure 1. Effects of mixing and mass transport.

TABLE 1. INITIAL RATE DATA FOR THE CARBON-STEAM REACTION

			Gas Composition					
			H_2O		H_2			
Run No	Mass of Virgin Carbon[1] $\times 10^6$, kg	Temp. K	$pH_2O \times 10^{-2}$ Pa	%	$pH_2 \times 10^{-2}$ Pa	%	Rate $\times 10^5$ R kg/(kg) (s)	Corr. for Oxygen Reaction % of Total Rate
9—S[2]	1.635	1003	3.02	0.298	0	0	0.285	44.1
10—V[3]	1.577	1002	3.00	0.307	0	0	0.327	40.2
11—S	1.508	1002	3.09	0.305	0	0	0.488	34.9
12—S	1.596	1003	3.11	0.307	0	0	0.378	40.9
13—S	1.710	1002	44.7	4.41	0	0	0.731	13.5
14—V	1.811	1031	0.720	0.0711	0	0	0.265	48.9
15—V	1.891	1031	1.50	0.149	0	0	0.513	40.8
16—S	1.528	1032	3.12	0.308	0	0	0.735	29.3
17—S	1.608	1033	3.00	0.296	0	0	0.705	44.1
18—S	1.998	1032	3.12	0.308	0	0	0.653	26.0
19—S	1.735	1032	3.06	0.302	0	0	0.828	23.7
20—S	1.280	1032	3.08	0.304	0	0	0.887	25.7
21—V	1.430	1032	4.39	0.433	0	0	0.937	29.3
22—V	1.480	1032	6.67	0.658	0	0	1.04	26.5
23—V	1.550	1032	10.6	1.05	0	0	1.06	25.2
24—S	1.428	1032	18.2	1.80	0	0	1.20	20.4
25—V	1.872	1032	28.9	2.85	0	0	1.26	19.0
26—S	1.729	1032	29.6	2.92	0	0	1.35	16.3
27—S	1.500	1032	45.4	4.48	0	0	1.52	14.7
28—S	1.827	1033	62.0	6.12	0	0	1.75	13.2
29—V	1.950	1061	0.796	0.0786	0	0	0.542	51.3
30—V	2.130	1061	1.50	0.148	0	0	1.21	32.0
31—S	1.705	1062	3.05	0.301	0	0	1.95	19.0
32—S	1.752	1061	3.05	0.301	0	0	1.68	22.8
33—S	2.039	1061	3.07	0.303	0	0	1.75	21.5
34—S	1.734	1061	3.34	0.330	0	0	1.60	7.9
35—S	2.425	1061	45.1	4.45	0	0	3.14	11.5
36—S	1.319	1091	0.386	0.0381	0	0	0.578	66.7
37—S	1.409	1091	0.778	0.0768	0	0	1.34	43.6
38—S	1.649	1091	1.38	0.136	0	0	2.38	27.8
39—S	1.916	1092	3.02	0.298	0	0	3.87	19.6
40—V	2.579	1091	3.13	0.309	0	0	3.70	16.2
41—V	1.420	1091	3.15	0.311	0	0	3.35	11.2
42—S	1.733	1091	44.7	4.41	0	0	7.33	9.9
43—S	1.677	1031	3.20	0.316	1.56	0.154	0.178	28.3
44—S	2.444	1061	2.93	0.289	1.42	0.140	0.523	24.0
45—S	2.302	1061	3.11	0.307	2.32	0.229	0.402	29.9
46—V	1.510	1090	3.20	0.316	3.00	0.296	0.828	32.3
47—V	1.610	1091	3.08	0.304	4.71	0.465	0.557	41.1
48—S	1.170	1120	3.01	0.297	1.71	0.169	2.55	27.8

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[1] Mass of virgin carbon unburned when samples reach reaction temperature.

[2] S designates B sample.

[3] V designates virgin carbon.

reaction of traces of oxygen in the gas stream. If the oxygen content could have been reduced to zero, these two straight lines should have been horizontal. The more rapid decrease in weight from $t = 0$ until the curves become straight lines is attributed to desorption of volatile matter from the virgin carbon. This is because all curves in Figure 2, with or without steam or for either virgin carbon or B samples, show the same transition region. Such losses in weight of virgin carbon was observed in Part I between 773 and 1002 K (Figure 6 of Part I) and attributed to the same cause.

After the transition period, the straight line sections of the curves V-steam and B-steam give the reaction rate for virgin and spent carbon. At larger time values (not shown in Figure 2) the slope decreased, presumably due to decreased surface area as large amounts of carbon were gasified. The weight change in the straight-line region is sufficient to restore spent carbon to its original adsorption capacity. This is evident from the nitrogen adsorption data presented later.

It is noted in Figure 2 that weight changes are very low (a few percent). Hence, care must be taken in correcting the measurements for very small changes in weight not due to the steam reaction. In our experiments these other changes were presumably caused only by traces of oxygen.

The rate of the steam-carbon reaction was evaluated from the straight-line sections of the curves for the steam runs by subtracting the weight decrease for runs with pure helium (for example, subtracting the weight decrease for run 7-S from that for run 11-S (Figure 2). This correction was made for each run by changing the gas back to pure helium after the run with steam had been completed.

In runs such as 11-S, both virgin carbon as well as residual carbon are gasified. The slope of the line for 11-S is essentially the same as that for run 13-V, where only virgin carbon is reacting.

The estimated time for mass transfer from gas to particle surface, based upon a stagnant layer (Sherwood number of 2), is 8×10^{-5}s. The comparable time for intraparticle diffusion was estimated in Part I to be 8×10^{-4}s at 500 K and would be less in the range 1003 to 1123 K. In comparison, the rate constant for the steam-carbon reaction at intermediate conditions suggests a time constant of the order of 10^3s. The rate of gasification with steam is a relatively slow reaction. From these estimates it is

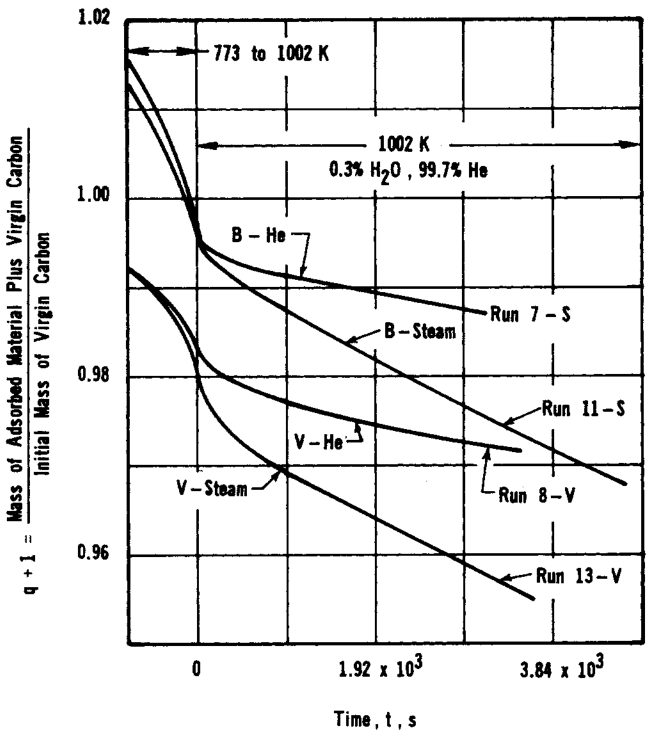

Figure 2. Mass vs. time for steam-carbon reaction.

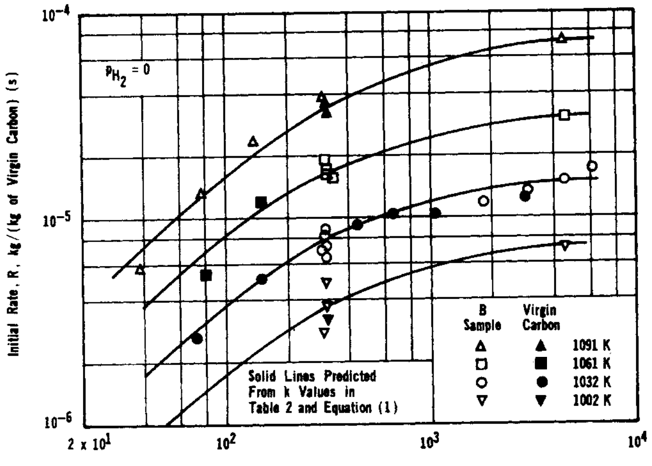

Figure 3. Effect of steam concentration on reaction rate.

concluded that weight vs. time data, such as illustrated in Figure 2, are a measure of the rate of the gas-solid reaction at the surface of the particle.

A summary of all the rate data is given in Table 1.

KINETICS OF STEAM-CARBON REACTION

Concentration Effects

The conversion of steam flowing over the sample was always less than 4%. Hence, the rates calculated from the weight-change data were initial values corresponding to the feed gas composition.

The data for $p_{H_2} = 0$ are shown by the points in Figure 3 for virgin carbon and for B samples. The results indicate a decrease from first order in H_2O to zero order as the partial pressure of steam increases. This observation applies at all four temperatures. The range of partial pressures of steam corresponds to a composition range of 0.04 to 6.0% since the total pressure was essentially 101 kPa (1 atm). These results agree with Eq. 1, as shown in Figure 4 where the data are plotted in accordance with the following linearized form:

$$\frac{1}{R} = \frac{k_3}{k_1} + \frac{k_2}{k_1 p_{H_2O}} p_{H_2} + \frac{1}{k_1} \frac{1}{p_{H_2O}} \tag{2}$$

The slopes and intercepts of the straight lines drawn through the data, which apply for $p_{H_2} = 0$, determine the values of k_1 and k_3 given in Table 2. The agreement of the data points for virgin carbon and for B samples in Figure 4 confirm that the rate of the steam reaction is the same for virgin and spent carbons. The

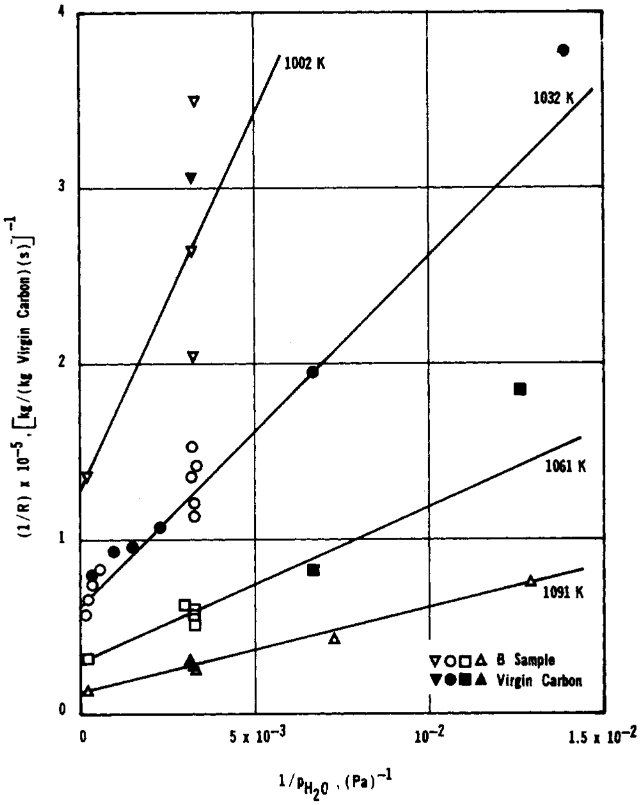

Figure 4. Linearized plot of rate data—effect of p_{H_2O}.

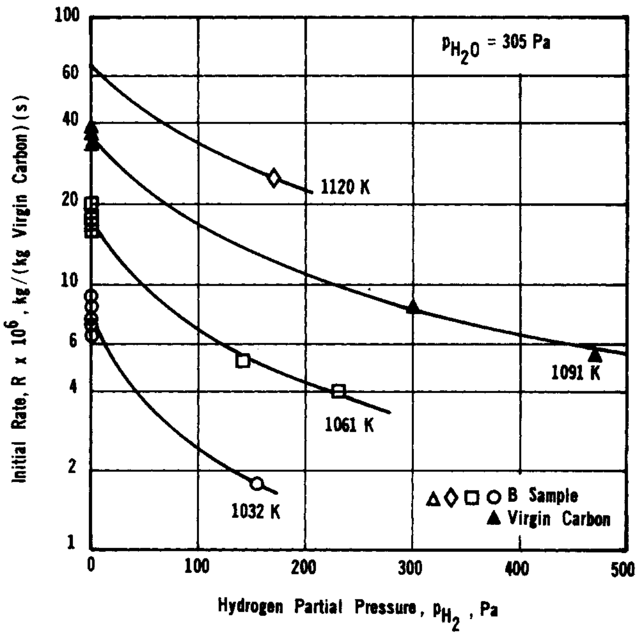

Figure 5. Effect of hydrogen concentration on reaction rate.

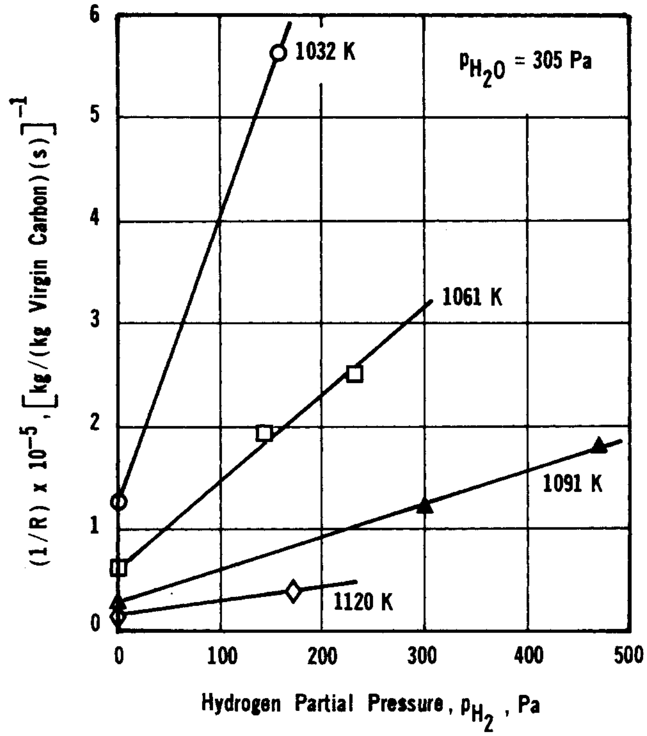

Figure 6. Linearized plot of rate data—effect of p_{H_2}.

curves in Figure 3 were drawn using the k_1 and k_3 values listed in Table 2.

The effect of hydrogen concentration on the rate, for a constant steam concentration (0.3%), is displayed in Figure 5, again for virgin carbon and for B samples. Even for the relatively small values of p_{H_2} (up to 0.5% in the gas) the retardation effect of hydrogen on the rate is significant. According to Eq. 2 a plot of $1/R$ vs. p_{H_2} would give a straight line whose slope is equal to $k_2/(k_1 p_{H_2O})$. The data points do follow this behavior as illustrated in Figure 6. The values of k_2, determined from the slopes of these lines, the known p_{H_2O}, and k_1 from Table 2, are also given in Table 2. The curves in Figure 5 were calculated using the k_1, k_2 and k_3 values in the table.

We conclude that Eq. 1 provides a satisfactory equation for steam gasification of BPL activated carbon, or spent carbon from sucrose decomposition, over the stated range of temperatures and gas composition.

Temperature Effects

As mentioned, there is not general agreement for the sequence of elementary reactions leading to the rate equation. If we regard Equation (1) as of the Langmuir-Hinshelwood form, k_1 would be interpreted in terms of an activation energy, while k_2 and k_3 might be regarded as adsorption equilibrium constants. The effect of temperature on these rate constants is shown in Figure 7. The behavior of k_1 does follow the Arrhenius equation with an activation energy of 2.27×10^5 J/mol. Also, k_2 varies with temperature in the direction expected from the van't Hoff equation; that is, with an apparent negative enthalpy of adsorption amounting to -1.02×10^5 J/mol.

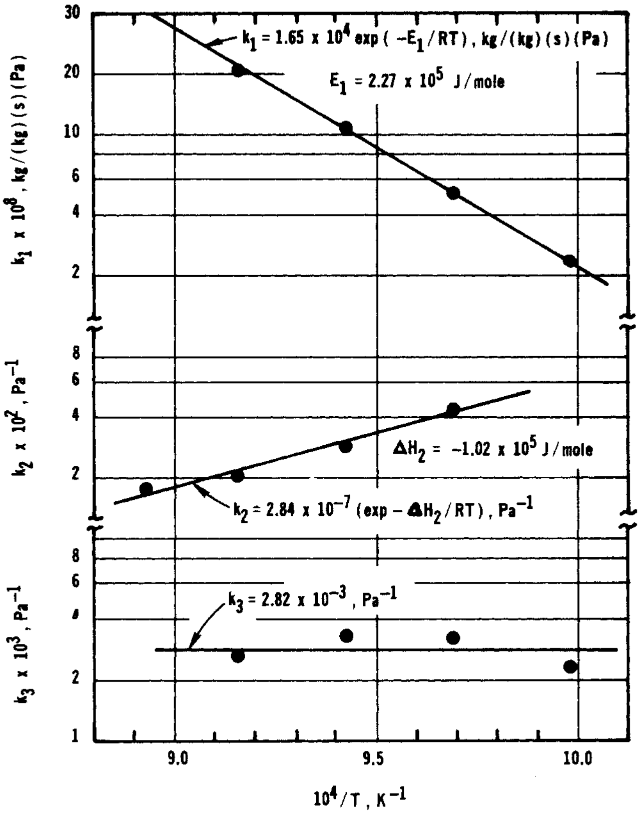

Figure 7. Effect of temperature on the rate constants.

The constant k_3 associated with H_2O in Eq. 1 does not vary significantly with temperature (Figure 7). The available data for the reaction of many types of carbon with steam, as reviewed by Walker, et al. (1959), suggest an overall effect of temperature corresponding to activation energies from 2 to 3×10^5 J/mol.

SURFACE AREAS AND PORE VOLUMES DURING REGENERATION

As noted in Part I, the decrease in mass adsorbed is a suitable measure of the extent of regeneration during the thermal process. However, the mass change during reaction with steam is not a reliable measure of regeneration because some virgin carbon is gasified. Surface area measured by nitrogen adsorption is a more reliable indication of adsorption capacity. Figure 8 shows a complete history of surface areas during the overall process. The samples studied in the steam-reaction region were prepared in the same way as described for the kinetic measurements. The reaction conditions were 1033 K, $p_{H_2O} = 305$ Pa (0.3%), $p_{H_2} = 0$. The abscissa in Figure 8 for the reaction region is the carbon gasified divided by the residual carbon at 773 K. In Part I it was found that the residual carbon at the end of thermal regeneration (i.e., q_{xh} at 773 K) averaged about 14% of the initial amount adsorbed (q_{oh}) for B samples (Table 2, Part I). Figure 8 demonstrates that the area of the regenerated samples reaches that of the virgin carbon when the carbon gasified is 220% of the residual carbon at 773 K. Thus at least 120% of the weight change is due to reaction of virgin carbon.

TABLE 2. KINETIC CONSTANTS FOR THE STEAM-CARBON REACTION.

Temp., K	1002	1032	1061	1091	1120
k_1, kg/kg · s · Pa	2.34×10^{-8}	5.08×10^{-8}	1.09×10^{-7}	2.08×10^{-7}	(4.10×10^{-7})*
k_2, (Pa)$^{-1}$		4.33×10^{-2}	2.83×10^{-2}	2.03×10^{-2}	1.75×10^{-2}
k_3, (Pa)$^{-1}$	2.25×10^{-3}	3.14×10^{-3}	3.27×10^{-3}	2.60×10^{-3}	(2.99×10^{-3})*

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* Extrapolated value.

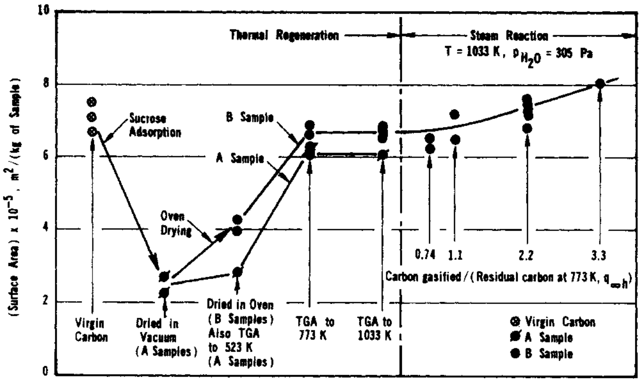

Figure 8. Surface area changes during regeneration.

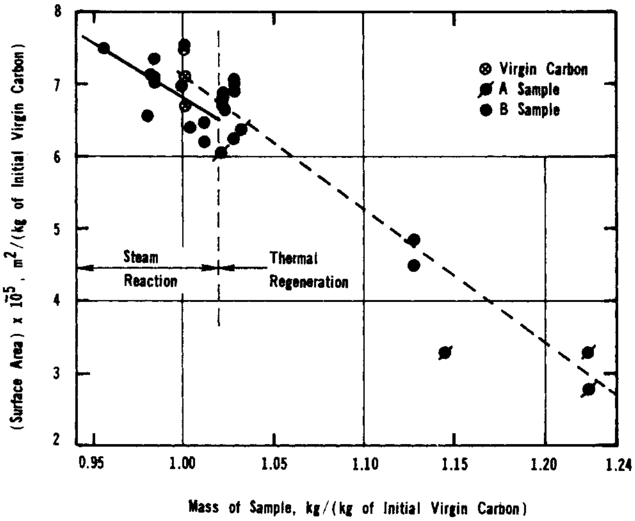

Figure 9. Surface area vs. weight change of sample.

The ordinate in Figure 8 is based upon a unit mass of sample. A more useful measure of regeneration is obtained by relating the area to that of the original virgin carbon. Figure 9 shows such areas plotted vs. the mass of the sample, corrected for volatilization of virgin carbon, per unit mass of initial virgin carbon. While the data in the reaction region scatter, two conclusions are possible: 1), the original adsorption capacity can be restored by reaction, and 2), the rate of regeneration of surface per unit mass of carbon is about the same as in the thermal process (the slopes of the solid and dotted lines are about the same). Also, the solid line for the reaction region is lower than that for thermal regeneration, since there has been some gasification of the virgin carbon. The type of data shown in Figure 9 for the reaction region is important for determining the optimum extent of the steam reaction to use in cyclical adsorption-regeneration cycles. As the extent of gasification with steam increases, the surface area increases, but also more of the initial virgin carbon is lost.

Pore volumes were also measured for samples at various stages of regeneration. The data obtained by nitrogen adsorption at a pressure of 49% of saturation (at liquid nitrogen temperature) gives the surface adsorption plus volume in pores of radius less than 2.2 nanometers. These results were similar to the area data in Figure 9. Presumably adsorption of sucrose would fill pores of small diameter. The regeneration of this pore volume was found to increase linearly with weight loss during thermal regeneration, as in Figure 9. During subsequent regeneration with steam, the pore volume increased further but at a somewhat lower rate.

ACKNOWLEDGMENT

Discussions with D. Misic and M. Suzuki were helpful in this research. The financial assistance of the National Science Foundation, Grant 77-15953 is gratefully acknowledged. We would like to thank the Calgon Corp. for providing activated carbon.

NOTATION (also see Part I)

E_1 = activation energy for rate constant k_1, J/mol
k_1 = rate constant for steam-carbon reaction, Eq. 1, kg/kg · s · Pa
k_2, k_3 = parameters in rate equation, Pa^{-1}
p_{H_2}, p_{H_2O} = partial pressures of hydrogen and steam, Pa
ΔH_2 = enthalpy of adsorption for hydrogen, J/mol
R = initial rate of steam-carbon reaction, kg of carbon reacted/(kg of virgin carbon unburned when the samples reach reaction temperature) (s)

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