FIRTH: THE SORPTION OF IODINE BY CARBONS, ETC.

XL.—The Sorption of Iodine by Carbons Prepared from Carbohydrates.

By James Brierley Firth.

The work of investigators on sorption by carbon has hitherto been mainly confined to carbons of either animal or vegetable origin, such as bone charcoal, blood charcoal, or the different varieties of wood and nut charcoal. The exception seems to have been sugar carbon, and to a lesser degree lampblack. It would appear that if carbons of different molecular complexity could be prepared, they would have a corresponding variation in activity and sorptive capacity.

The object of this series of investigations is to determine, if possible, whether the complexity and nature of the original compound influence the activity of the carbon produced from it, and also to determine the influence (if any) of such elements as nitrogen, sulphur, and the halogens in the parent substance. It is therefore proposed to prepare carbons from pure substances belonging to the various classes of organic compounds.

The present investigation is confined to carbons prepared from carbohydrates.

EXPERIMENTAL.

The chloroform was dried for several days over calcium chloride and fractionated, the middle portion only being taken.

Resublimed iodine was employed as solute.

The carbohydrates used were the purest obtainable, and those marked K were Kahlbaum's preparations. The carbohydrates were carbonised at as low a temperature as possible and the conditions were as nearly as possible the same in all cases. sulting carbon was finely powdered, digested with concentrated hydrochloric acid, washed with water, and finally boiled with water until free from chloride, and the purity of the carbon then determined. Cellulose carbon was prepared from the best surgical cotton wool. It was digested successively with dilute potash, dilute hydrochloric acid, water, alcohol, and ether to remove admixtures and finishing materials. It was then carbonised as in other cases. A specimen of sugar carbon was prepared by adding concentrated sulphuric acid to a concentrated sugar solution, the resulting carbon was repeatedly digested and washed with boiling water until free from sulphate, and then treated as in other cases. The approximate density of each carbon was determined by the water displacement method. Although the values for density are not to be regarded as absolute, the figures indicate satisfactorily the relative densities of the various carbons.

N/10-Solutions of iodine in chloroform were used throughout. The carbon was heated to a dull red heat, out of contact with the air, immediately before being used. One gram of the finely divided carbon was used in each case and treated with 25 c.c. of the N/10-iodine solution. The experimental details were very similar to those already stated (Firth, $Trans.\ Faraday\ Soc.,\ 1921,\ 16,\ 434$).

TABLE I.

Carbon prepared from	Percentage ash as prepared.	Percentage ash after purification.	Density.	Remarks.
Sucrose by heat	0.245	0.086	1.501	$\left\{egin{array}{l} ext{Bright;} \ ext{easily} \ ext{powdered} \end{array} ight.$
$\left. egin{array}{l} \operatorname{Sucrose} \\ \operatorname{by} \ \operatorname{H}_2 \operatorname{SO}_4 \end{array} ight\}$	0.773	0.189	1.700	**
Glucose	1.784	0.512	1.502	,,
Maltose (K)	0.111	0.098	1.509	"
Lactose (K)	0.637	0.372	1.619	
Lævulose (K)	0.323	0.129	0.410	,,
Wheat		0.461	1.434	∫ Dull; easily
starch }	0.504	0.401	1.434	powdered
Potato } starch	2.810	1.326	1.515	**
Rice starch	2.770	1.036	1.436	Fine black powder, easily pyrophoric
Dextrin (K)	0.661	0.432	1.497	$\left\{egin{array}{l} ext{Bright;} \ ext{easily} \ ext{powdered} \end{array} ight.$
Inulin (K)	2.071	1.117	1.506	Very hard; difficult to powder
Cellulose	0-486	0-301	1.480	Very fine powder, easily pyrophoric

The temperature of experiment was 18° . The results are expressed in terms of 100 c.c. of solution; the mass of carbon, m, = 4 grams; x/m = grams of iodine sorbed by 1 gram of carbon; a-x = the final concentration of the solution in grams per 100 c.c. The time of exposure of the carbon to the solution was varied from five minutes to seven days. The results are given in Table II.

From Table I, the purest carbon of the series is seen to be that prepared from maltose, whilst potato starch carbon finally contained the highest percentage of ash. With three exceptions, the ash was considerably less than 1 per cent., whilst in two cases it was less than 0·1 per cent. During carbonisation, most carbohydrates passed through a very viscous phase and gave rise to a friable, metallic-looking carbon mass. In the cases of rice starch and cellulose, no such phase occurred, and the ultimate product was a very fine, easily pyrophoric powder, whilst inulin gave rise to a dull and exceedingly hard carbon.

From Table II it will be observed that the various carbons differ very much in *activity*. The values of x/m for an interval of five minutes vary from 0.06672 in the case of levulose carbon to 0.2917

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TABLE II.

Source of	NOTAL CO OL						
carbon.	Time.	x/m.	a-x.	carbon.	Time.	x/m.	a-x.
	6 mins.	0.2439	0.2941		c 5 mins.	0.1562	0.6450
	30 ,,	0.2540	0.2537	Dextrin	30 ,,	0.1647	0.6110
Sucrose (by heat)	2 hours	0.2622	0.2209		2 hours		0.5486
	24 ,,	0.2847	0.1309		2 hours 24 ,,	0.1981	0.4774
	3 days	0.2958	0.0865		3 days	0.2149	0.4102
	7 ,,	0.3085	0.0357		7 ,	0.2363	0.3246
i	5 mins.	0.1859	0.5261	Cellulose	5 mins.	0.2911	0.1050
	30 ,,	0.1961	0.4853		30 ,,	0.2942	0.0930
Sucrose] 2 hours	0.2136	0.4153		2 hours	0.2963	0.0846
$(by H_2SO_4)$	24 ,, 3 days	0.2289	0.3541	Centilose	24 ,, 3 days	0.3006	0.0674
	3 days	0.2631	0.2173		$3 ext{ days}$	0.3073	0.0406
,	7 ,,	0.2762	0.1649		7 ,,	0.3106	0.0274
	6 5 mins.	0.2917	0.1019		7 5 mins.	0.2858	0.1265
	30 ,,	0.2954	0.0881		30 ,,	0.2892	0.1129
Chance	2 hours	0.2979	0.0781	Potato	2 hours	0.2931	0.0973
Glucose	2 hours 24 ,,	0.3012	0.0649	starch	24 ,,	0.2969	0.0821
	3 days	0.3092	0.0329		3 days	0.3047	0.0509
,	7 ,,	0.3104	0.0281		7 ,	0.3098	0.0305
Maltose {	$\begin{cases} 5 \text{ mins.} \\ 30 & ,, \\ 2 \text{ hours} \\ 24 & ,, \\ 3 \text{ days} \end{cases}$	0.1951	0.4893		5 mins.	0.2346	0.3313
	30 ,,	0.2025	0.4597		30 ,,	0.2483	0.2765
	2 hours	0.2142	0.4129	Rice	2 hours	0.2587	0.2349
) 24 ,,	0.2429	0.2981	starch	24 ,, 3 days	0.2692	0.1929
	3 days	0.2868	0.1225		3 days	0.2878	0.1185
	(,,	0.3012	0.0649		7 ,,	0.2998	0.0705
$\mathbf{Lactose} \ \left\{ \right.$	$\begin{cases} 5 \text{ mins.} \\ 30 & ,, \\ 2 \text{ hours} \\ 24 & ,, \\ 3 \text{ days} \\ \end{cases}$	0.1734	0.5762		5 mins.	0.2301	0.3494
	30 ,,	0.1841	0.5334		30 ,,	0.2456	0.2874
	1 2 hours	0.1952	0.4890	Wheat		0.2532	0.2570
	24 ,,	0.2270	0.3618	${f starch}$	24 ,,	0.2699	0.1902
	3 days	0.2680	0.1978		3 days	0.2868	0.1226
	7 ,,	0.2996	0.0714		7 ,	0.2987	0.0750
Lævulose	5 mins.	0.06672	1.0029		5 mins.	0.1698	0.5906
	30 ,, 2 hours 24 ,, 3 days 7 ,,	0.07854	0.9556	Inulin	30 ,, 2 hours 24 ,, 3 days	0.1827	0.5390
	J 2 hours	0.09364	0.8952		2 hours	0.2001	0.4694
	24 ,,	0.1328	0.7386		24 ,,	0.2192	0.3930
	3 days	0.1545	0.6518		3 days	0.2352	0.3290
	7 ,,	0.1741	0.5734		7 ,,	0.2581	0.2374

in the case of glucose carbon, and for twenty-four hours the values are 0·1328 and 0·3012, respectively. It will be observed from Table I that lævulose has the lowest density of the series, but the results in general do not indicate that density is a primary factor in determining the activity of the carbon. Glucose, cellulose, and potato starch carbons show very high activity, and with the exception of lævulose the remainder of the carbons are of medium activity.

After a period of seven days, the total iodine sorbed by the respective carbons appears to be approaching an identical value; that is, ultimately the sorptive capacity in all cases will be the same. In order to test this view, the three carbons from glucose,

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lævulose, and dextrin, under the same conditions as before, were exposed to the iodine solution for six months; the x/m values then were: glucose 0·3128, lævulose 0·2893, dextrin 0·2962. These results would indicate, therefore, that under the conditions of the present experiments the ultimate sorptive capacities of the carbons of this series are the same.

Summary.

For the conditions of the experiments as herein described:

- 1. The activity of the carbons prepared from carbohydrates may vary over quite a large range.
- 2. The greatest activity is displayed by the carbon prepared from glucose, and the least by the carbon prepared from lævulose.
- 3. The ultimate sorptive capacity of all the carbons prepared is the same.

THE CHEMICAL DEPARTMENT, UNIVERSITY COLLEGE, NOTTINGHAM.

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