

XLI.—*A Chemical Method for preparing Carbon Hydrosol.*

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IN the course of an investigation on the catalytic decomposition of aqueous solutions of sodium hypochlorite, it was found that charcoal acts very vigorously. The action is not, however, a purely catalytic one, for the charcoal is oxidised with the formation of carbon dioxide. It was noticed that when the charcoal was treated repeatedly with solutions of the hypochlorite, the liquid acquired a yellowish-brown colour and evidently contained a carbon (or possibly carbonaceous) sol. In most of the experiments described in this paper, a purified form of charcoal was employed, which had been boiled with concentrated hydrochloric and hydrofluoric acids and repeatedly washed with water until its ash content (mainly

silica) was reduced to 0.2—0.3%. The charcoal had also been heated to 900° in a high vacuum and had been used in experiments on the adsorption of oxygen gas, by which means any adsorbed hydrogen was removed.

0.3 G. of the charcoal was placed in a flask and 100 c.c. of distilled water were added; 5 c.c. of sodium hypochlorite solution (prepared from chlorine gas and alkali solution, and approximately  $M/4$  with respect to  $\text{NaClO}$ ) were poured into the flask and the mixture was rapidly stirred in a thermostat at 25°. Similar amounts of the hypochlorite solution were added at intervals of 2—3 hours. The concentration of the hypochlorite solution is immaterial, but in order to obtain a concentrated sol, a fairly large amount of hypochlorite is necessary. As the increasing concentration of sodium chloride is prejudicial to the stability of the sol, it is best to stop the operation from time to time and remove the sodium chloride by dialysis before adding more hypochlorite. It is also advisable to start with small amounts of charcoal and gradually work up the concentration of the sol. The colloidal solution thus obtained is now dialysed until it is as free as possible from extraneous electrolytes. In this condition, it reacts neutral to the usual reagents and is very stable (no deposition was observed after several months). It can be evaporated on the water-bath without undergoing any perceptible change, and if the operation be carefully conducted, the residue passes back into colloidal solution on the addition of water. The colour of the dilute solution is yellow by transmitted light, and that of the concentrated sol, viewed in a thin layer, varies from burgundy-red to dark brown. The colloid is not destroyed on being boiled with concentrated nitric acid. If a little of the sol be treated with a few c.c. of concentrated hypochlorite solution, the carbon particles are gradually completely oxidised and a clear colourless solution is obtained. The solid residue obtained by evaporating the dialysed sol at 100° loses about 20% of its weight on prolonged heating at 300°, and this loss is probably due mainly to the removal of adsorbed water.

Cataphoresis measurements showed that the sol particles are negatively charged (moving towards the positive electrode with a velocity of  $70 \times 10^{-6}$  cm./sec. under a gradient of 1 volt per cm.). It is probable that the negative charge is due to the adsorption of chlorine (and possibly hydroxyl) ions. This assumption is supported by the high precipitating power of the univalent silver ion (see following table).

*Coagulation by Electrolytes.*—The coagulating power of various electrolytes has been measured, and the results are summarised in the following table.

## Coagulation of carbon sol by electrolytes.

Electrolyte.	Limiting value (millimols./litre).	Relative coagul- ating power.	Degree of precipitation.
NaNO <sub>3</sub> .....	2500	0.4	Not complete.
AgNO <sub>3</sub> .....	9.1	110	Complete.
LiCl .....	1180	0.84	Not complete.
NaCl .....	1000	1.0	" "
KCl .....	552	1.8	" "
CaCl <sub>2</sub> .....	2.6	384	" "
SrCl <sub>2</sub> .....	1.6	625	" "
BaCl <sub>2</sub> .....	1.15	865	Complete.
Ba(NO <sub>3</sub> ) <sub>2</sub> .....	1.3	770	" "
AlCl <sub>3</sub> .....	0.86	1163	Not complete.
Al(NO <sub>3</sub> ) <sub>3</sub> .....	1.1	909	Complete.
Th(NO <sub>3</sub> ) <sub>4</sub> .....	0.6	1540	" "
NaOH .....	682	1.46	" "
Ba(OH) <sub>2</sub> .....	4.5	222	" "

These results can only be regarded as rough approximations, as the minimal values were not always well defined. They confirm the cataphoresis experiments, and show that the sol contains particles stabilised by the adsorption of negative ions.

*Size of Particles.*—On the assumption that the carbon particles possess the density of graphite (2.16), an ultramicroscopic examination of a sol containing 0.325 g. of dry residue per litre gave 60  $\mu\mu$  for the average radius of the particles. Another sol containing 0.88 g. of dry residue per litre gave 68  $\mu\mu$  for the average radius. Some of the particles in these sols must be very small, for a double membrane of collodion and parchment paper did not prevent the diffusion of a certain amount of the sol. For this reason attempts to measure the osmotic pressure did not give constant and steady results. It may be remarked, however, that relatively high values for the maximum observable pressure were obtained. Thus, in the case of a sol containing 0.325 g. of dry residue per litre, the maximum osmotic pressure, observed at 25° after several changes of the outside water, was equal to 20 cm. of water.

*Carbon Ink.*—If a dialysed sol of carbon, prepared in the manner described in this paper, be evaporated on the water-bath, a brownish-black, thick liquid is obtained which can be used as an ink. The writing has a sepia colour and is resistant to washing and to the action of chemical agents. This fact may possess some technical value, as it was also found possible to prepare these stable carbon sols from unpurified commercial charcoal.

*Theory of Formation of the Carbon Sol.*—Although a chemical examination of the dry residue obtained from the sols described in this paper is reserved for a future communication, it is suggested that the particles consist essentially of carbon, and that the formation of the sol is due to the gradual breaking down or "etching" of

the surfaces of the particles of charcoal (caused by chemical oxidation to carbon dioxide) whereby particles possessing vigorous Brownian motion are produced, these being stabilised by the adsorption of chlorine (or hydroxyl) ions derived from the hypochlorite solution.

*Summary.*

1. A carbon hydrosol has been prepared from purified and from unpurified charcoal by the oxidising action of solutions of sodium hypochlorite.

2. The well-dialysed sol is very stable and can be evaporated at 100° without irreversible coagulation. It exhibits all the properties of a finely dispersed negative sol.

3. From these sols a good, brown, carbon ink can be obtained.

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