ORIGINAL CONTRIBUTIONS

A RAPID QUANTITATIVE METHOD FOR DETERMINING THE DECOLORIZING POWER OF CARBONS

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In the examination of various decolorizing carbons for water soluble and acid soluble content, ash, specific gravity, etc., it was found necessary to have some method that would give a comparison of the decolorizing power of the same carbon and of different decolorizing carbons. As all the various quick methods had an indefinite end-point, due to being masked or due to lag, iodine was considered as a solute in that it had a definite end-point for titration and could be determined quantitatively. The methods in use are dye methods and color measuring methods.

The dye methods depend on the use of a definite weight of some dye, as 2.0 g. of methyl violet dissolved in 600 cc. of alcohol and diluted to 2000 cc. with water. One gram of the carbon is weighed into a 300 cc. beaker and 50 cc. of water is added. The methyl violet solution is run into the beaker slowly from a buret and the solution is vigorously stirred. The appearance of a purple tinge in the foam indicates the end-point. This end-point should be permanent for two to five minutes. The result is expressed in terms of the amount of carbon required to decolorize 0.05 of methyl violet. A second method is the use of 0.05 g. of eosin scarlet in 1000 cc. of water. The decolorizing power is expressed by the number of cc. of eosin scarlet that are decolorized by 0.3 g. of carbon.

The dye methods are very inaccurate in that the end-point is covered by the presence of the carbon in the solution, the end-point is not permanent in the presence of the carbon and the size of particle is not taken into consideration.

The methods for measuring color are well presented by Sanders,[1] in that the color of the particular solution to be treated by a chosen carbon is measured for color before and after carbon treatment. These methods require a color-measuring instrument, which in most cases is not a part of the equipment of the ordinary laboratory. Also, the preparation of the carbon and the manipulation of the solution requires considerable time before the sample is ready for color measuring.

The work of J. W. McBain[2] and J. B. Firth[3] on adsorption of iodine in a solvent of benzene or chloroform offered a possible means to determine the decolorizing power of carbons by treating a standard aqueous solution of potassium iodide and iodine with carbon, and determining by means of a

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[1] *Chem. & Met. Eng.*, **28,** No. 12.

[2] *Jour. Chem. Soc.*, **91,** p. 1683.

[3] *Trans. Faraday Soc.*, **16,** p. 434.

standard sodium thiosulphate solution the quantity of iodine adsorbed. This method as developed must be understood to express the decolorizing value of carbons for iodine solution, or its "Iodine Number."

The solutions used in this method are:

I. 0.2 *N* iodine-potassium iodide solution (iodine, 25.384 g. and 50.0 g. KI per liter).

II. 0.1 *N* sodium thiosulphate ($Na_2S_2O_3.5H_2O$, 24.832 g. per liter).

III. 0.1 *N* tassium dichromate ($K_2Cr_2O_7$, 4.903 g. per liter).

IV. Starch solution (25.0 g. per liter).

The sodium thiosulphate solution was standardized against 0.1 *N* $K_2Cr_2O_7$ and the iodine by the 0.1 *N* sodium thiosulphate. The method as developed for the determination of the decolorizing power is as follows.

The Method

A sample of carbon ground through a 300-mesh sieve is dried for five hours at a temperature of 105°C. Five-tenths gram of the sample, weighed accurately to within 1 mg., is placed in a 1 liter beaker and 490 cc. of distilled water and 10.0 cc. of iodine solution are added in the order named. This solution is stirred for 3 minutes and filtered through absorbent cotton. Two hundred and fifty cc. of the filtered solution is caught in a volumetric flask as a sample for titration with the 0.1 *N* thiosulphate solution, using, near the end-point, 0.5 cc. of starch solution and titrating to the first disappearance of the blue color. A blank is now run in the same way but using no carbon. The decolorizing power for iodine can be calculated by subtracting the $Na_2S_2O_3$ titration of the sample from that of the blank, e.g.,

$$\frac{\text{cc. } Na_2S_2O_3 \text{ equivalent to iodine adsorbed} \times 100}{\text{cc. } Na_2S_2O_3 \text{ equivalent to total iodine used (blank)}} = \%\text{ iodine adsorbed}$$

or the decolorizing value.

The proper weight of carbon to be used was determined by keeping all the variables constant and only varying the weight of carbon. As different

TABLE 1

VARIATION WITH WEIGHT OF DECOLORIZING POWER

		Carbon Number			Carbon Number	
Weight Carbon Grams	1	2	3	1	2	3
		Cc. $Na_2S_2O_3$			Per cent decolorizing power	
0.050	7.45	7.90	6.75	11.84	6.52	20.05
.100	6.45	7.55	5.50	23.70	10.65	34.95
.175	5.05	7.20	3.90	40.30	14.80	53.80
.250	4.10	6.50	2.80	51.50	23.07	66.90
.375	2.55	5.60	1.55	67.50	33.70	81.66
.500	1.50	4.75	0.75	82.30	43.75	91.20
.675	0.85	3.80	.30	89.90	55.00	96.45
.800	.55	3.25	.15	93.50	61.50	98.25
1.000	.30	2.50	.10	96.50	70.45	89.90
.....	8.45	8.45	8.45			

Time, 3 minutes. Samples unprepared for size.

weights adsorbed different amounts of iodine, the results so obtained when plotting weight of carbon against per cent of iodine adsorbed show the "break" at the point where the value of the carbon axis is 0.5 g. (Table 1).

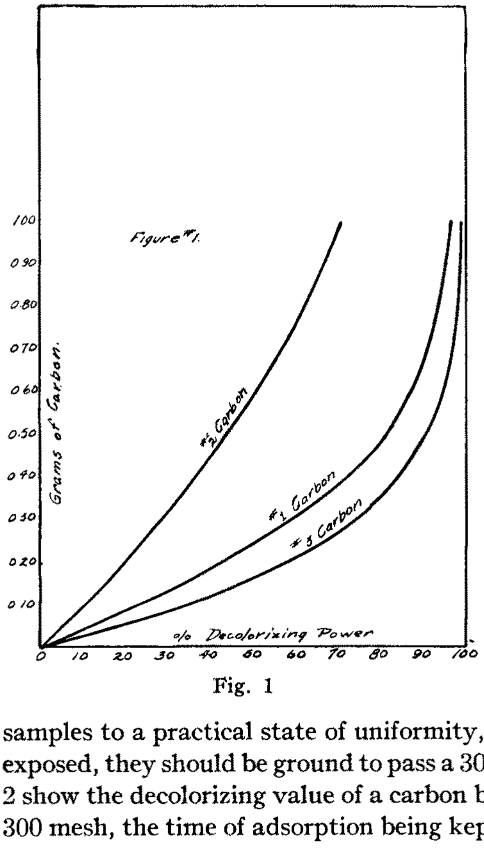

Fig. 1

It is evident that the curves tend to straighten out at the ordinate 0.5 g. and thereafter follow very closely the vertical ordinate. Firth[1] also pointed out this fact when he plotted log $\frac{x}{M}$ and log $(a\text{-}x)$ as derived from the adsorption equation of Freundlich, $\frac{x}{M} = BC\,\frac{1}{p}$, where x is the weight of the material adsorbed, c is the concentration after treatment, and B and p are constants.

By plotting the foregoing results according to the logarithmic equation, the curves from the ordinate 0.5 g. are straight lines. The value a is the weight of the total amount of solute used.

The size of the particles making up the sample is important, as the size of the particle determines the surface exposed for adsorption. To bring all the samples to a practical state of uniformity, relative to the amount of surface exposed, they should be ground to pass a 300-mesh sieve. The data in Table 2 show the decolorizing value of a carbon before and after grinding through 300 mesh, the time of adsorption being kept constant at 3 minutes.

TABLE 2

VARIATION WITH SIZE OF DECOLORIZING POWER OF NO. 1, CARBON

Weight Carbon	Unground	300 mesh	Unground	300 mesh
Gram	Cc. $Na_2S_2O_3$		Per cent	Decolor. power
0.500	2.40	1.75	77.66	82.50
.500	2.90	2.00	70.56	80.00
.500	2.65	1.90	73.09	81.00
.500	3.20	2.05	67.51	79.50
.500	3.30	2.60	66.49	74.00
.500	2.50	1.95	74.62	80.50
....	9.85	10.00		

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[1] Firth, *Loc. cit.*

Naturally if one is using the same carbon at all times and wishes to check up the relative decolorizing values of various lots from the manufacturer, a fairly good comparison can be made without reducing the samples to a uniform size.

In order to determine the time for iodine adsorption the weights were varied and the time of agitation held constant for 3 minutes, 6 minutes and 9 minutes. The time data in Table 3 show a very small and practically constant difference in the rate of the thiosulphate titration after the second 3-minute agitation period.

O. C. M. Davis[1] states that the surface condensation is nearly instantaneous and is quite complete in a few hours: the diffusion (capillary absorption) proceeds for weeks, months and even years.[2] To check our results on "time," the 3-minute period was overrun a minute with and without stirring, no change being found in results.

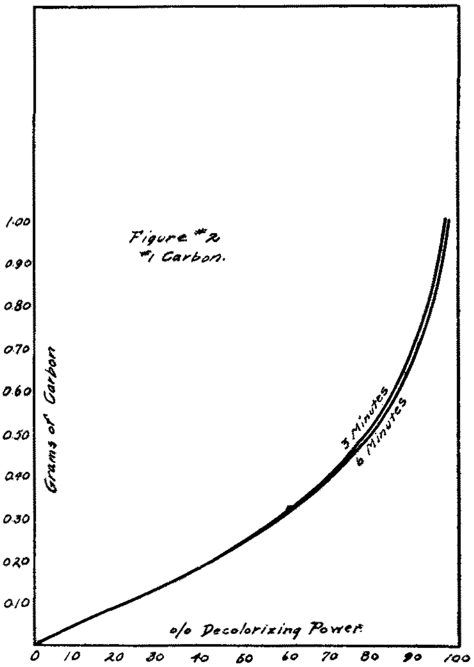

Fig. 2

The temperature is not important if the entire manipulation is carried on at room temperature, as the running of the blank allows for the correction of

TABLE 3

VARIATION WITH TIME OF DECOLORIZING POWER, OF NO. 1 CARBON

Weight Carbon Gram	Minutes Treated 3	Minutes Treated 6	Difference in $Na_2S_2O_3$ titrations	Minutes Treated 3	Minutes Treated 6
	Cc. $Na_2S_2O_3$			Per cent Decolor. power	
0.050	8.80	8.55	0.25	10.66	10.94
.100	7.80	7.60	.20	20.81	20.83
.175	6.20	6.00	.20	37.05	37.50
.250	5.00	4.80	.20	49.24	50.00
.375	3.50	3.30	.20	64.77	65.62
.500	2.05	1.95	.10	79.19	79.69
.675	1.15	1.00	.15	88.32	89.59
.800	0.75	0.60	.15	92.39	93.75
1.000	.30	.20	.10	96.95	97.92
....	9.85	9.60	.25		

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[1] *Jour. Chem. Soc.*, **91**, p. 1666.

[2] *Loc. cit.*

any small amount of iodine that will volatilize. Nevertheless, dispatch in manipulation should be exercised to cut any possible loss to a minimum, due to the volatility of the iodine. From the initial weighing of the sample to the final titration, a maximum time of 10 minutes should suffice.

Conclusions

1. This method is a rapid quantitative method for the determination of the decolorizing power of carbons for iodine.
2. The method can be used for controlling the plant output of any one kind of carbon without a size preparation.
3. The method can be used for comparing decolorizing carbons manufactured by different methods.
4. The adsorption for iodine is practically instantaneous.

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A NEW TYPE OF COLOR-COMPARATOR

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History of Oil Colorimetry

Not so many years ago, the determination of the color of oils, in the cottonseed oil industry, was of minor importance, but when this factor was introduced into the trading specifications it soon became necessary to have some basis upon which to report colors. The earliest of these standards was probably bichromate of potassium solutions made up in definite concentrations. It was soon found, however, that such solutions did not simulate the color of refined cottonseed oils sufficiently to prevent grave disagreements between the buyers' and sellers' analysts.

The next step was perhaps the adoption of the Lovibond glasses and these are still in almost universal use, by American oil chemists. They have not, however, proven entirely satisfactory. Much time and energy have been given by members of the American Oil Chemists' Society, to finding some means for bringing about better agreement between their color readings. These studies have followed two distinct lines; the development of a new type of color comparator or a simple spectrophotometer; and refinements in, or more specific directions for, the use of the Lovibond glasses. Dr. Priest of the U. S. Bureau of Standards has most generously aided in devising a new color instrument and it is largely as a result of his work, that the E-K Color Analyses described elsewhere in this issue of the JOURNAL, has been placed on the market. Dr. Wesson in 1920 enlisted the assistance of the Eastman Kodak Company in the problem and they built, for a number of oil laboratories, a very convenient type of color comparator. This instrument is quite expensive and has the added disadvantage that its