Decolorizing Carbon Analyses and Tests

Describing in Detail Methods for Evaluation of the Quality and Effectiveness of Bleaching Carbons

In Two Parts—Part I

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THE methods described in this article are those in use in the factory and experimental laboratory of a large manufacturer of decolorizing carbons. In compiling this paper no attempt has been made to include every conceivable test of carbons, but only those tests which will be of help to the user of carbons and which will aid him in making an intelligent selection of a carbon for his purpose. The author will welcome any suggestions for additions or improvements.

Moisture

WEIGH out approximately ten grams of the carbon into a crystallizing dish fitted with a cover. Remove the cover and place the dish in an electric oven. Dry overnight at 140° C. (Drying at 105° C. does not remove the last trace of moisture.) Place the dish and its cover in a dessicator to cool. When cold, replace the cover on the dish and weigh the dish, cover and carbon. The loss in weight is taken as representing the moisture present.

Calculation:—

$$\frac{\text{Loss in weight of carbon} \times 100}{\text{Weight of carbon taken}} = \%\ \text{moisture}$$

It is necessary to cover the dish containing the sample as the dried carbon absorbs moisture very rapidly. Save this sample for the determination of water soluble matter.

Screen Test

A THOROUGH separation of carbon into the respective mesh is possible only by water screening. The procedure is as follows:

Size of Sample.

A convenient size of sample is between 7½ and 10 grams of carbon.

Preparation of Sample.

Place the sample of carbon of known dry weight, in a liter beaker and wet the sample with water, taking care to avoid the loss of dust. Fill the beaker about two-thirds full of water. Stir the mixture. Thorough wetting of the carbon is necessary to the ease of separation on the screens.

Preparation of the Screens.

Before being used, the screens must be cleaned, inspected for holes and thoroughly wet.

When screening with water, small particles of carbon lodge between the wires of the screen and are not readily removed by passing water through the screen from either side. These particles may be removed by lightly brushing the screen with a test tube brush and at the same time washing the screen with a gentle stream of water from a laboratory hose.

Stack the wetted screens in the same manner as for dry screening, but with the top cover and bottom pan omitted. Apply water to the top screen and note whether it passes readily through the screens.

Transfer of the Carbon to the Screens.

The "slimes" (the very fine carbon which floats on the water) give the most trouble, as it is practically impossible to thoroughly wet this fine material. Practically all of the "slimes" will pass the 300 mesh screen, and it is well to be rid of them at the beginning of the test. Accordingly, let the sample in the beaker settle. Pour the top part on the top screen and wash the carbon through the screens, using a gentle stream of water from a hose.

If the entire sample, even with the "slimes" removed, were put on the screen at once the lower screens (of finer mesh), would become plugged and the water would not pass through. Unless this condition is noticed at once, the plugged screen will overflow and the test will be ruined.

For this reason, transfer the sample to the screens slowly, passing a gentle stream of water

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through at the same time. It is necessary to watch the amount of carbon collecting on the 200 and 300 mesh screens. If the sample be added too rapidly the fines will not have time to be washed through these small mesh screens, but will cover these screens and flood them.

When the sample has been transferred to the screens, wash out, with the hose, any particles of carbon which may remain in the beaker. Continue to add water to the top screen until no particles of carbon are seen coming out of the bottom screen.

Determination of completeness of separation.

Place the screens over a large, white porcelain evaporating dish and collect, in this dish, the water passing through the screens. Any particles of carbon which pass the screens can readily be seen on the white surface.

This method, however, does not show absolutely that the separation is complete, when a stack of screens are used, as the water may channel. Accordingly, when the washing of a stack of screens seems complete, it is necessary to take each individual screen and test it in the same manner.

To do this, start with the top screen, wash it with the hose and collect the water in a large evaporating dish. If it contains carbon, put this water with the carbon through the remainder of the stack. Continue this procedure until the water from this screen comes free from carbon particles. Repeat this method with each screen. When all the screens have been thoroughly washed, the carbon is ready to be transferred for weighing.

Transfering of various mesh carbon for weighing.

By means of a stream of water from the hose, transfer the carbon remaining on each screen, into separate beakers. Transfer the carbon in each beaker into a separate tared Gooch crucible fitted with an asbestos mat. Dry the crucibles and carbon in an oven at 140° C. Cool them in a dessicator and weigh them. The weighing must be done rapidly as the dried carbon absorbs moisture readily.

The net weight of carbon in each Gooch crucible times 100, divided by the dry weight of the original sample, gives the percent of carbon of each size.

The amount of carbon passing the 300 mesh screen is taken by difference.

The screens usually employed are of 100, 200 and 300 mesh. However, one can use the screens which will give him the information he seeks.

Apparent Density

THE apparent density of a carbon is determined by measuring the volume occupied by a known weight of carbon when packed so that it will not decrease in volume by continued tamping, or by measuring the amount of carbon which may be packed in a container of known volume.

Determine the approximate weight of *dry* carbon necessary to fill a 100 c.c. graduated cylinder to the top graduation. Weigh out this quantity of dried carbon and transfer it to the cylinder. Stopper the top of the cylinder to avoid loss of dust and fine particles of carbon. Tamp the carbon to the minimum volume which it will occupy, by tapping the cylinder on a table until no further settling occurs. A large rubber stopper will make a good buffer between the table and the bottom of the cylinder.

Read the volume occupied by the carbon. The apparent density is equal to the grams of dry carbon taken, divided by the volume, in c.c., occupied by the packed carbon.

Oil Saturation Value (O. S. V.)

THE O. S. V. of a carbon is expressed as the grams of cotton seed oil required to wet 100 grams of carbon.

The test is carried out as follows:—

A small weighed sample of carbon (about 5 to 10 grams) is placed in a 250 c.c. porcelain casserole. The clear cotton *seed* oil is added drop by drop. After each addition, the carbon is stirred gently, care being taken to see that all traces of oil have disappeared before adding more oil. The carbon mass will ball, either forming one or a few large lumps, or remain in smaller lumps which, however, will adhere together towards the end. The character of the mass makes no difference as long as no oil is added while traces of it are still visible. The end point is reached when the casserole shows the first trace of streakiness. This becomes quickly apparent, for previous to the addition of the last few drops, the surface of the casserole becomes perfectly clean and bright.

The oil saturation value is expressed as grams of oil per 100 grams of dry carbon. If the oil has been added from a burette the volume is known and knowing the specific gravity of the oil, the O. S. V. is readily calculated.

The O. S. V. of a carbon is a function of the apparent density. After the section entitled, "Oil Retention Value," will be found Table I. which gives the "Apparent Density," "Oil Saturation Value" and "Oil Retention Value" of practically all grades of various commercial decolorizing carbons and a discussion of these properties.

Oil Retention Value (O. R. V.)

THE oil retention value is a measure of the oil held in the cake of a filter press after blowing the press. It is expressed as grams of oil per 100 grams of carbon.

The O. R. V. shown in Table I. were determined as follows:

One hundred grams of carbon (dry basis) were mixed with four liters of refined, unbleached cotton seed oil and heated to 50-55° C. This mixture was then filtered through a laboratory plate and frame press which had been pre-heated by blowing steam through it for ten minutes before filtering. 30-40 pounds pressure were used for filtering the carbon-oil mixture. After filtering, the press cake was treated as follows:

1. Cake blown with air for 15 minutes at 40 lbs. pressure.
2. Cake blown with steam for 15 minutes at 30 lbs. pressure
3. Cake blown with air for 10 minutes at 40 lbs. pressure.

The cake was removed from the press, thoroughly mixed and the oil retained in the cake was determined by extraction.

TABLE I.

Carbon Number	Apparent Density Grams per C.C.	Gms. oil per 100 gms. Carbon O.S.V.	O.R.V.
1	0.245	258.0	105.0
2	0.252	249.0	98.4
3	0.270	220.0	105.0
4	0.275	222.0	93.0
5	0.288	183.0	72.4
6	0.292	185.0	71.4
7	0.295	204.0	75.2
8	0.310	179.0	70.3
9	0.320	175.0	68.3
10	0.390	148.0	39.0
11	0.485	125.0	36.1
12	0.515	118.0	33.0
13	0.540	110.0	29.0
Bone Char	1.00	50.8	18.3

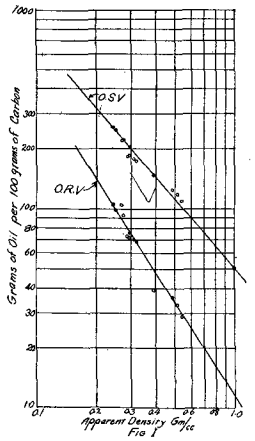

Fig 1

If the logarithm of the O. S. V. be plotted against the logarithm of the apparent density, it will be found that the points fall nearly on a straight line. Likewise, a logarithmic plot of the O. R. V. against the apparent density gives a straight line. These curves are shown in Figure I.

We see from an inspection of these curves that a carbon of high apparent density will retain a much smaller quantity of oil in the press cake than will one of a low apparent density.

Filtrability

FILTRABILITY tests made on a Buchner funnel do not mean a great deal. The best way to test the filtrability of a carbon is to filter a full size works' batch in the works' filter press. If this is undesirable, some idea of the filtrability of a carbon may be gained by the use of a laboratory filter press, for instance, one with 6 inch frames, offering ¼ square foot of filtering area per filter cloth.

When testing two carbons for filtrability the same pressure and temperature should be used in each case. This pressure and temperature should be approximately that used on the large presses in the plant. The rate of flow of the filtrate should be determined. One should also note the time elapsing between the start of the liquor through the press and the time at which the filtrate becomes clear.

The rate of filtration is ordinarily reported as gallons of filtrate per square foot of filtering surface per hour.

Water Soluble Matter

TAKE the ten gram sample which was used for the moisture determination and transfer it to a 600 c.c. tall form beaker, with lip. Add 200 c.c. of distilled water. Heat on an asbestos center gauze until the mixture begins to boil, stirring continually during the time of heating. Remove the flame and stir the contents of the beaker for five minutes. Allow the carbon to settle and then decant the clear, supernatant liquor through a tared Gooch crucible fitted with an asbestos mat.

Repeat the digestion three times, each time adding a 200 c.c. portion of distilled water to the residue in the beaker.

After the fourth digestion, transfer the carbon to the crucible, suck the mass as dry as possible and then dry the crucible and contents over night, in an electric oven, at 140° C. Cool the crucible and contents in a dessicator and weigh.

$$\frac{100\ (\text{dry weight of carbon—wt. after extraction})}{\text{dry weight of carbon}} = \%\ \text{total water soluble matter. (dry basis.)}$$

Experiments have shown that more than four extractions are not necessary, as even sixteen extractions do not increase the result by as much as 0.05% water soluble matter.

Ash

THE ash is determined as follows:—Two grams of carbon of known moisture content, are weighed out into a porcelain crucible 42 m.m. top diameter and 35 m.m. depth. The crucible is then placed in an oven and dried at 140° C. for several hours. After drying, the crucible is placed on a Nichrome triangle and ignited with the flame of a Meker burner, cooled in a dessicator and weighed. The ignition is repeated until the loss in weight after 15 minutes ignition amounts to less than 1 mg.

$$\%\ \text{Ash (dry basis)} = \frac{\text{dry weight of carbon taken}}{100 \times \text{weight of ash}}$$

It will be found that if the carbon be ignited directly after weighing out the sample, without the preliminary drying in the electric oven, the moisture or adsorbed air will be suddenly liberated, throwing part of the carbon from the crucible.

The ash is a great aid in the identification of the source of a carbon, as the appearance of the ash of various brands of carbon is quite characteristic. Scarcely any two of them have the same color and form. It so happens that when the color and structure of the ash from two carbons are similar, the percentages of ash differ widely.

Method of Determining the pH of a Carbon (Acidity or Alkalinity)

THE term "pH of a carbon" is somewhat misleading. It is in reality the pH of water which has been in contact with a carbon under certain specific conditions. The pH will vary with the ratio of water to carbon used, the time of contact of the water with the carbon and the temperature of water while the carbon is in it. The following was decided upon as standard.

Weigh 100 grams of dry carbon (or its equivalent of moist carbon) into a 2 liter balloon flask of Pyrex glass, cover with 1 liter of freshly boiled distilled water, fit the flask with a 15″ reflux condenser (Liebig) and boil for 1 hour. (In case there are acid or ammonia fumes present in the laboratory, fit the top of the condenser with a cork stopper and a piece of glass tubing that will extend outside of the laboratory.) At the end of this period remove the flame, allow the carbon to settle and then by means of a 20 ml. pipette with an extension, remove through the top of the condenser a small sample of the supernatant liquid while it is still hot (90° C.). Remove the extension from the pipette, cool the sample under the tap and transfer it to the hydrogen electrode vessel and determine its pH in the usual way or transfer 10 c.c. of it to a small test tube and determine its pH colorimetrically.

The electrometric method is considered the standard method for the determination of pH while the colorimetric method is only a quick and easily operated substitute method. The latter method gives the results accurate to within ±0.2 pH units in ordinary cases, but in the range just below the neutral point the accuracy is considerably less, due to small amounts of acid or alkali and lack of buffer salts. The errors, however, should be reasonably constant, and with careful work and good manipulation the results should be comparative.

The materials required—Lamotte color standards (contained in sealed glass ampoules) and corresponding indicator solutions, covering the pH range desired, several test tubes with 10 c.c. graduations and of same glass and diameter as ampoules, and several 1 c.c. pipettes with mark to deliver 0.5 c.c.

Procedure—If the sample (water solution referred to above) contains carbon, filter it through a small filter paper into another 10 c.c. test tube. Add to the clear solution 0.5 c.c. of the indicator solution covering the pH range which the solution is suspected to be within, stopper the test tube with a small rubber stopper, invert the test tube a few times and then match the color obtained with one of the color standards which is made up with 10 c.c. of the buffer solution and .5 c.c. of the same indicator. Estimate the pH to the nearest tenth of a unit.

Precautions must be taken to prevent the access of carbon dioxide as far as this is possible, especially after the solution is filtered. The work should be carried through rapidly, and the tubes kept stoppered. This is particularly necessary when the sample is nearly neutral.

The most important point is to have proper lighting for matching colors. It was found that for solutions without color or turbidity, an ordinary test tube rack with a sheet of filter paper in back of it is satisfactory, furnishing an even, white background.

The color standards should be protected from heat and direct sunlight. With the exception of the set covering the very low acid range, they are fairly permanent. Those sets

covering the pH range 3 to 9.6 should be checked up at least once a year.

The method outlined above for the determination of the pH of a carbon has been thoroughly tested in the laboratory of the Darco Corporation and has been found to give consistent results. Equilibrium is so slowly reached that it is necessary to boil the mixture of carbon and water for one hour. At room temperature, equilibrium is so slowly attained that the results will not become constant even after many hours of shaking the mixture.

Decolorizing Ability of a Carbon

IT HAS been our experience that the removal, by carbons, of color from liquids follows Freundlich's adsorption equation. This equation is:

$$X/M = KC^{1/n} \quad \text{.....(1)}$$

Where X = amount of Solute adsorbed,
M = grams of carbon used,
C = concentration of Solute after adsorption.
K and 1/n are constants.

Since we are ordinarily interested in the color of the solution rather than the actual concentration of the colored substance, we use certain "color units" in place of the concentration terms X and C of Freundlich's equation.

It is known that the transmission of light of any given wave length (color) through a colored solution is according to the following equation, provided that the colored substance does not undergo chemical change with change in concentration.

$$I/I_0 = e^{-kdm} \quad \text{.....(2)}$$

Where I = the intensity of the light emerging from the solution.
I_0 = the intensity of the light entering the solution.
e = the base of natural logarithms.
k = a constant.
d = the distance which the light travels through the solution.
m = the concentration of the colored substance in the solution.

If we let $I/I_0 = T$

Where T = the fraction of incident light transmitted by the solution—we can re-write equation (2) as follows:

$$m = \frac{-\log_e T}{kd} \quad \text{.....(3)}$$

Therefore, in place of C and X in equation (1), we can use any system of color units in which the color units are proportional to the logarithm of the fraction of light transmitted.

Equation (2) holds only for monochromatic light. The eye is affected by all colors. The effect on the eye of the color of a substance is measured by the area under the curve obtained by multiplying the energy values of the light of each wave length by their relative abilities to produce light sensation (luminosity). (See Luciesh—Color and Its Applications," published 1915 by D. Van Nostrand Co., New York, pages 35-36, 209-210.) Such a scheme is followed by the Bureau of Standards in their method of measuring the color of sugars where the spectral transmission curves of the different sugars vary somewhat. The Bureau of Standards' system of color measurement, as applied to sugars, is described in "Sugar," May 1925, pages 223-224. The Bureau of Standards also uses a function which they designate as "Q" which is the ratio of the negative logarithm of the transmission of light of a given wave length to the negative logarithm of the transmission of light of 560 millimicrons. This "Q" ratio is useful in studying the effect of various reagents and treatments on sugar solutions. The Bureau of Standards' color unit is based on a sugar concentration of one gram per cubic centimeter and a layer one centimeter thick. For our present purpose of comparing the degree of activation of two carbons, we do not need to use the Bureau units, although it would be well to do so in order to follow a standard system.

In the example given later in this manual, we reduce the color units to the basis of a layer of solution one centimeter thick, but as we use a molasses solution, we do not reduce the color units to a basis of one gram of solids per cubic centimeter of solution.

At the Darco Laboratory the system of color units described in "Chemical and Metallurgical Engineering," March 21, 1923, page 541 is still in use. We intend to continue using this system until the method of measuring the color of solution is standardized, at which time a change in method will probably be made.

This last named system is a modification of that of Meade and Harris described in "Industrial and Engineering Chemistry," Vol. 12, page 687 (1920). Meade and Harris' system was thus modified in an attempt to have the color units more nearly represent the color of the solution as it affects the human eye.

(To be Concluded)

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It is said that the difference in iodine number of the unsaponifiable matter in olive oil and that in the oils commonly used as its adulterants can be used in determining adulteration in amount of 5% or less. The iodine number of the unsaponifiable is 197-206 in olive oil and 117-124 in the adulterating oils. *Olii minerali, olii e grasi, colori e vernici,* 11,9-10 (1931). *Chem. Abstr.* 24,4140 (1931).

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In a recommended process for the saponification of liquid soaps, boiling in two stages is advocated. The difficultly saponifiable fats are boiled first; the remainder of the charge is added later, and next day enough olein is added to bring the alkalinity to 0.1 per cent, after which enough softened water to produce the desired concentration is added. *Seifensieder-Ztg.* 58,340-1 (1931).