CHARCOAL FROM PINE CONES

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CHARCOAL FROM PINE CONES.

The value of vegetable charcoal as a preservative and a deodorizer was undoubtedly well known to the ancient Egyptians and Romans. One of the embalming methods of the Egyptians was to wrap the body in charcoal. Foundations which were built in wet marshy grounds, were placed on a layer of charcoal, which it was thought would afford a protection from the moisture. Food for animals was thought would be kept from decaying by the addition of pieces of charcoal.

Although charcoal was made use of in very many ways its special properties remained for a long time unknown.

In 1786, Lowitz,[1] an apothecary in St. Petersburg, in attempting to prepare a large quantity of tartaric acid, found difficulty in obtaining pure white crystals, in as much as the solution became brown due to charring during evaporation. In one experiment, more charring than usual took place and he set the whole mass aside not knowing what to do with it. Upon examining it the next day he was surprised to find that the charred substance had

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1. Crell's Ann. d Chem., 1786, I, p. 217.

settled to the bottom leaving a colorless solution above from which he obtained pure white crystals. He at once attributed the decoloration of the tartaric acid solution to the charcoal and showed that with the aid of vegetable charcoal he could at all times prepare white crystals of tartaric acids as well as other organic compounds. He also succeeded in decolorizing such things as vinegar, fruit juices, linseed oil and honey.

In 1791 Lowitz,[1] published a second series of experiments on the decolorizing power of charcoal. Immediately after Lowitz published his results, many other men tried the decolorizing effect of charcoal, but in most cases obtained negative results. For examp

says:

"After a long series of experiments I have thoroughly convinced myself that Lowitz is in the wrong as much as I wish that the mistake was on my part."

In 1791, Kels,[3] apparently unaware of any previous work on this subject, gives an account of the decolorizing power of vegetable charcoal. He decolorized solutions of saffron, indigo, and other pigments, also sugar solutions.

Dubuqua,[4] in 1803, published an article on the decol-

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1. Crell's Ann. d Chem., 1791, p. (Ann. de Chim. (1) 14, p. 327.
2. Arch. der Pharm., 14, p. 114.
3. Gren's Jour. d Physik, 4, p. 33.
4. Ann. der Physik, Gilbert, 13 p. 103.

orizing power of vegetable charcoal, in which he says that the experiments were performed without any knowledge of the work of Lowitz. It was not until about to publish his results that he learned of the earlier work of Lowitz. The fact that the water could be purified by vegetable charcoal, led him to perform a great number of experiments on the action of charcoal on different substances. He prepared the charcoal from "Weidenholz" and succeeded in decolorizing wine, indigo solutions, honey, oils, etc. He also prepared seven different colored solutions, corresponding to the colors of the spectrum and found that the red solution was decolorized at the end of ten or twelve days and the violet at the end of forty days and the other colors according to their position in the spectrum.

Figuier, in 1811, was the first to suggest the use of animal charcoal as a decolorizing agent in the place of wood charcoal. In working on a method for decolorizing vinegar, he recommended the use of animal charcoal, not only as giving better results, but also a saving of time and material. He found the decolorized vinegar to contain small amounts of calcium acetate and phosphate, which came from the charcoal used. He therefore recommends that the charcoal be first purified by boiling with HCl, Sp. Gr. 170 Baume. This is then the origin of the U. S. P. method of purifying animal charcoal.

About the same time it occurred to Payen,[1] who manufactured ammonia by the destructive distillation of bones that the charred residue might be used in the refining of sugar. With this end in view he sent some of the powdered carbon to the Imperial beet sugar factory at Rambouillet. From a great number of experiments made at this place, the conclusion was arrived at that the animal charcoal could not be used for sugar refining on a large scale. Two years later Derosne and Pluvinet[2] made a great number of experiments regarding the relative decolorizing value of animal and vegetable charcoal, especially in the refining of sugar, and communicated their results to Payen. Payen then so modified the process of refining sugar by means of animal charcoal, that it was not long before the consummation of charcoal for this purpose in Paris alone was greater than could be supplied by Payen's two ammonia factories at Grenelle and Chichy.

The use of charcoal in the arts became of such importance that a prize was offered for the best paper on this subject by the Societe de Pharmacie de Paris.

Bussy, whose paper won the first prize,[3] determined the composition of both crude and purified animal charcoal and also the relative decolorizing value of different kinds of charcoal. The charcoal from dried blood was found the

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1. Jour. de Pharm., 14, p. 278.
2. Ibid 279.
3. Ibid 257.

most efficient.

Payen,[1] who won the second prize, arrived at practically the same conclusions as Bussy. He devised an optical instrument for determining the relative decolorizing power of the different charcoals.

Schrader,[2] in 1826, recommended a mixture of eight parts of dried blood and one part of potassium carbonate, which is heated to redness and the soluble parts washed out with water.

Dumont,[3] in 1828, invented a filtering apparatus to be used for decolorizing sugar with animal charcoal.

Stenhouse,[4] in 1856, prepared a decolorizing charcoal by heating 92.5 parts of vegetable charcoal with a solution of 7.5 parts of potassium phosphate and 20 parts of HCl in 40 parts of water. After boiling this mixture the charcoal was dried and heated. The resulting product had a decolorizing power equal to that of animal charcoal.

Because of the high price of animal charcoal Stenhouse,[5] in 1857, proposed several substitutes for it. The following is the process employed: 54 parts of sulphate of alumina were dissolved in water and digested with 92.5 parts of finely powdered wood charcoal. When the

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1. Jour. de Pharm., 14, p. 278.
2. Arch. d Pharm., 16, p. 103.
3. Jour. de Pharm. 21, p. 543.
4. Arch. d Pharm., 142, p. 57.
5. Pharm. Jour., 16, p. 363.

charcoal had been thoroughly saturated with the solution of sulphate of alumina, the mass was evaporated to dryness and heated to redness. Aluminized charcoal can be used to decolorize acid as well as other solutions quite as efficiently as animal charcoal and is much cheaper.

As a substitute for animal charcoal for decolorizing purposes, Facilides[1], in 1870, recommended the following: Dry vegetable charcoal in small pieces is moistened with Dippel's oil, and the mixture heated gradually in a shallow dish, provided with a cover, until a red heat is reached. After cooling and pulverizing a fine charcoal is obtained. No experiments are recorded relative to its decolorizing power.

Graeger[2], prepares a very active animal charcoal as follows: Commercial animal charcoal is boiled for some time with 4-6 parts of a 4 to 5 % solution of soda, and is then allowed to settle, which required from three to four days. The supernatant liquid is then decanted, the residue is mixed with a like amount of hot water and again allowed to settle. By this treatment the sulphates are decomposed and removed, which if present to a considerable extent interfere with the proper purification. After the waste water has been decanted, the charcoal is transferred

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1. Am. Jour. Pharm., 191, p. 138.
2. Proc. Am. Pharm. Assc., 21, p. 283.

to a large porcelain vessel and treated with an excess of HCl and water, using sufficient acid to prevent the formation of a precipitate upon the addition of a little ammonia water to a portion of the filtered liquid. A large quantity of spring water is now added, the charcoal is allowed to settle, the supernatant liquid is decanted, and the residue is washed several times with acidulated spring water. Finally, it is collected upon a filter, thoroughly washed with distilled water and dried at a temperature of 100° to 120° C. (212° - 248° F). 100 parts of crude yield 20 parts of dry purified animal charcoal if the form of an exceedingly light, soft, black powder of very superior decolorizing property.

Schwarz[1], in 1873, found that animal charcoal can be entirely revivified in closed vessels by ighition with organic matters, such as glue, sugar, etc.

Melsens[2] produced an artificial decolorizing charcoal, equal to bone black, by impregnating wood charcoal with phosphate of calcium dissolved in HCl. The mass thus prepared is ignited and then washed with water.

According to Mueller[3], 1891, a vegetable charcoal can be prepared by carefully heating wood and allied substances

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1. Chem. News, 26, p. 194.
2. Ibid 1874, p. 165.
3. Proc. Am. Pharm. Assc., 39, p. 499.

with strongly alkaline solutions under great pressure. The product is equal if not superior and cheaper than charcoal obtained from blood. After use it can easily be regenerated by treatment with HCl, ignited and thoroughly washed with water. It has been very satisfactory in clarifying and purifying water, organic and pharmaceutical preparations, oils, honey, glycerine, ammonia, tannin, vaseline, alcohol, etc.

METHODS OF DETERMINING DECOLORIZING VALUES OF CHARCOALS.

Two general methods of determining the decolorizing power of charcoal are used; one a chemical method, the other an optical method.

Schober,[1] in 1873, recommended the following method:

A solution of six grammes of indigo carmine is dissolved in enough water to make a liter of solution. A second solution is made having one gramme of potassium permanganate in a liter of solution. The comparative strength of these two solutions is determined by decolorizing 10 cc of indigo solution to which has been added sulphuric acid with the potassium permanganate solution.

One gm. of charcoal to be tested is put into a stoppered flask with 50 cc of indigo solution and allowed to stand with occasional shaking for 24 hrs., the solution is filtered through a dry filter and the amount of potassium permanganate solution required to decolorize 10 cc of the filtrate, is determined.

If the original carmine solution required 8.8 cc of potassium permanganate solution to decolorize it, and after treating with charcoal it required 4.3 cc of permanganate solution, then the charcoal destroyed as much

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1. Zeitsch. f Reuben Zucker Ind. 1873, p. 42.

color as would have been destroyed by 8.8 - 4.3=4.5 cc of permanganate solution. The percent of color destroyed by the charcoal can be calculated by the proportion,

$$8.8 : 100 ;; 4.5 : X$$

$$X=51.1\%$$

In 1886, Laube,[1] recommended the following method to be used especially in sugar factories:

Prepare a solution of 50 gms. of caramel in 100 cc of water, add 100 cc of alcohol and dilute the whole to one liter. After allowing to stand several days, filter. This is called the normal color solution. To determine the decolorizing coefficient of good animal charcoal proceed as follows:

5 gms. of charcoal are heated to boiling with 200 cc of water, 10 cc of normal color solution is added and the whole boiled gently for exactly 10 minutes with a reflux condenser and then filtered into a Nesslerizing tube. Into a second tube 200 cc of water is placed and then the normal color solution added drop by drop until the solution is of the same color as that of the filtered solution. If 2.1 cc of normal color solution was added, than 10 - 2.1= 7.9 cc of color solution was decolorized by the charcoal.

Besides these chemical methods, different optical in-

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1. Pharm. Central. 27, p. 615.

struments have been invented by Payen,[1] Stammer[2] and others for determining the relative decolorizing power of charcoal. For a detailed description the references can be consulted.

From time to time, vegetable charcoals from different sources have been suggested as a substitute for animal charcoal, both for medicinal and commercial purposes.

The hard, brittle nature of the cones of the American pine tree, led to the suggestion that a charcoal might be prepared from them, having special merits over other charcoals. The cones for this work were obtained from Dr. C. Shepperd, Pine Hurst, South Carolina.

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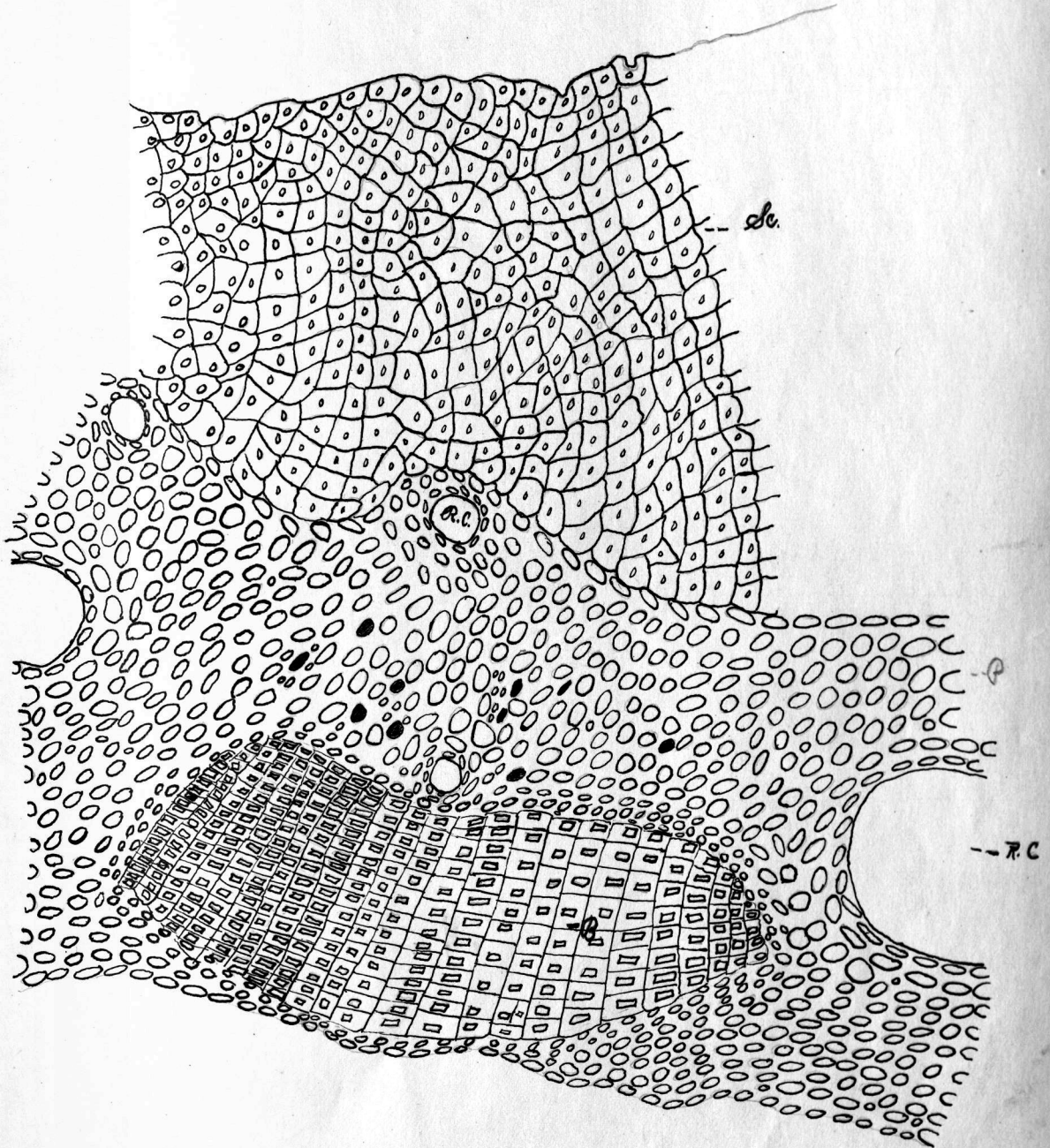
1. Polyt. Jour. 27, p. 372; Muspratt's Chem. 4, p. 1382.
2. Muspratt's Chem., 4, p. 1382.

CROSS SECTION OF SCALE OF CONE.

A cross section of one of the scales was made near the apex. When viewed with a microscope it appears as shown in Fig. 1.

The upper half consists of the sclerenchyma sheath composed of thick-walled cells, diameter of average cell wall being about .039 mm. The lower half of the section is made up of parenchyma tissue and the fibervascular bundle. The former contains the resin cells which vary in diameter from .045 to .234 mm.

The compactness of the cells, and the great thickness of the cell wall as compared with the diameter of the cell itself, would indicate that a very fine charcoal ought to result.



R.C. - Resin ducts.

Sc. - Sclerenchyma sheath.

B. - Fibro-vascular bundle.

P= Parenchyma.

DESTRUCTIVE DISTILLATION OF CONES.

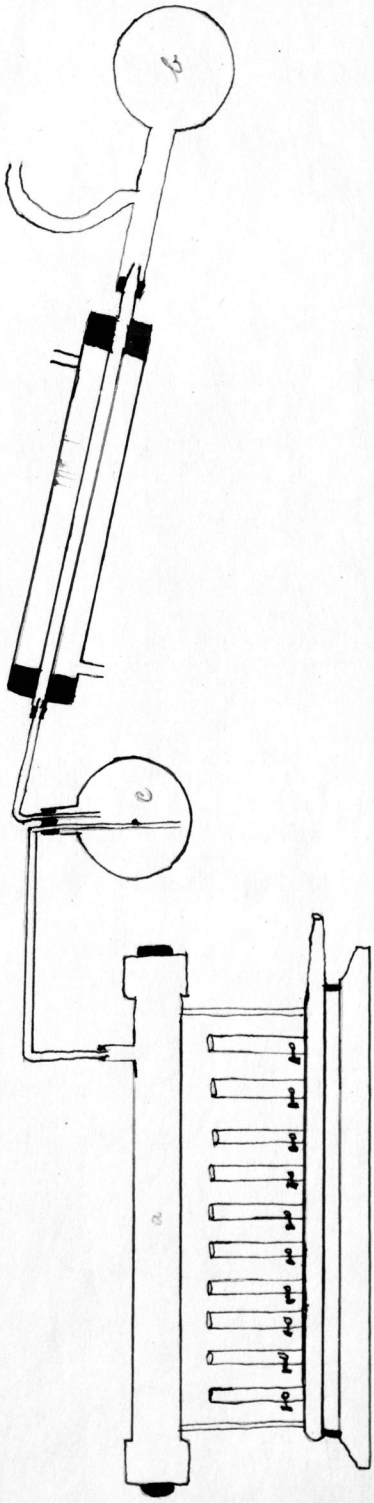
The cones were subjected to destructive distillation in an apparatus, as shown in the following diagram. 100 gms. of the cones, in small pieces, were put in the iron cylinder "a," and after the proper connections had been made, the burners were gradually lighted. The aqueous liquid was collected in flask "d," while most of the tar remained in flask "c." The gas was collected and measured in a gasometer after being passed through a strong solution of potassium hydroxide.

The following table shows the results obtained from the distillation:

TABLE NO. I.

	Wt.of subst.	Time of distillation	Wt.of tar	Wt.of aqueous liquid	Wt.of carbon	Vol. gas
I.	100 gms.	1 hr. 15 min.	10.62	37.45	43.6	2.5 L.
II.	100 "	30 "	7.3	29.99	36.3	4.6
III.	100 "	2 " 2 "	7.2	32.7	38.7	0.
IV.	100 "	1 " 10 "	5.95	28.3	37.0	1.5
V.	100 "	1 " 57 "	5.3	26.85	42.0	1.4

[illegible] and a greater quantity of charcoal was [illegible] istillation was allowed to go on slowly.


b
c
a

The charcoal obtained in each case was ground up in a drug mill, and then pulverized still finer in a mortar. The resulting fine powder, after exposure to air until no more odor was perceptible, was of a dull black color, was hard, gritty, practically tasteless. The charcoal from the different distillations all had about the same appearance. It was rather difficult to pulverize it, repeated pulverization in a mortar being necessary before a powder was obtained which was at all comparable in fineness with that of some commercial charcoals.

Inasmuch as the scales of the cones seemed to contain considerable resin, a considerable volume of combustible gas was looked for in the destructive distillation. In all cases, however, a very small volume of gas was obtained. See Table I.

A resin determination was therefore made to find out the exact percent. of resin which the cones contain. 100 grams of the pine cones were ground up in a drug mill to a No. 60 powder, and extracted with ether in a Soxhlet extraction apparatus. That part of the powdered material which did not pass through the No. 60 sieve, was extracted separately. The total ethereal extract was allowed to evaporate and the residue weighed. 7.05 gms. of resin were obtained corresponding to 7.05%.

DECOLORORIZING POWER.

The decolorizing power of the finely powdered charcoal from the pine cones was tested in comparison with that of other charcoals, as follows: A solution of 0.1 gram of Aniline violet in 1 liter was made and 10 cc. of this solution diluted to 100 cc. Each cc. of this solution therefore contained .00001 grams of aniline violet. A weighed quantity of the charcoal to be tested was put in a small beaker, and about 25 cc. of the color solution added. This was heated to boiling and gradually more solution, until upon filtering, the filtrate remained slightly colored. To determine the time required by each charcoal, the same quantity of charcoal was weighed out, and an amount of solution added as was formed in the previous experiment, the weight of charcoal used would just decolorize. The time required by the different charcoals was found to be approximately the same in each case, about 4 minutes.

In the following table are given the comparative decolorizing values of charcoal from pine cones, willow charcoal, and animal charcoal, as determined by the above method:

KIND OF CHARCOAL	WT. OF CHARCOAL	NO. OF cc. OF ANILINE VIOLET SOL.	TIME
1.Pine cone	2.1 gms.	100 cc.	
2. " "	1.05 "	50 "	4 minutes
3. " "	1.05 "	50 "	4 "
4.Willow	1.00 "	250 "	---------
5. "	1.00 "	250 "	4 "
6.Animal	1.00 "	140 "	
7. "	0.50 "	70 "	4 "

Experiments 2 and 3 were made with charcoals of different degrees of fineness. This seemed to have no effect on the amount of solution decolorized. Inasmuch as this method of determining the relative decolorizing values of the charcoals was rather unsatisfactory, a standard method, as recommended by Schober,[1] 3 grams of Indigo Carmine were dissolved in enough dilute sulphuric acid to make 500 cc. of solution. 10 cc. of this solution required 2.5 cc. of potassium permanganate solution, (1 gram in 1 liter). 1 gram of each of the charcoals to be tested was put in a small flask and 50 cc. of the indigo carmine solution added. The mixture was allowed to stand for 24

1. Zeitschr. f. Rübenzucker Ind. 1873, p. 42. (See Methods of Determining Decolorizing Values of Charcoals, p.)

hours with occasional shaking, and then filtered. The excess of indigo carmine in 10 cc. of the filtrate was titrated back with the permanganate solution. The percent of color destroyed by the charcoal was calculated as shown on page 9 of this thesis. The following tabulation shows the results obtained by this method:

KIND OF CHARCOAL	WT. OF CHARCOAL	cc. OF CARMINE SOL. ADDED	cc. of $KMnO_4$ SOL. REQ. FOR 10cc. of FILTRATE	% DECOLORIZED
--------	------	10 cc.	2.5 cc.	100.pc.
Pine cones	1	50 cc.	2. cc.	20.pc.
" "	1	50 cc.	2.1 cc.	16.pc.
" " (fine)	1	50 cc.	1.8 cc.	28.pc.
Pine cones (fine	1	50 cc.	1.85cc.	26.pc.
Willow	1	50 cc.	1.5 cc.	40.pc
"	1	50 cc.	1.5 cc.	40.pc.
Animal	1	50 cc.	.0	decolorized
"	1	100 cc.	.5 cc.	80.pc.

ALUMINIZED CHARCOAL.

So-called aluminized charcoal was prepared from the charcoal obtained from the pine cones, according to the directions given by Stenhouse.[1] 54 parts of aluminum sulphate were dissolved in water. 92.5 parts of the finely powdered charcoal were digested with this solution until the charcoal had become thoroughly saturated with the sulphate solution. The mixture was then evaporated to dryness, the dry mass put into a crucible and heated to redness in a muffler, until all the acid and water had been dissipated. The charcoal thus prepared became exceedingly fine upon pulverization in a mortar, and was of a dull black color. Upon testing its decolorizing value, according to Stober's method, 96% of the color of the indigo solution was destroyed in two duplicate experiments.

From the results obtained it can be seen that the charcoal prepared from pine cones is inferior as a decolorizing agent to both willow and animal charcoals. The willow and animal charcoals were much more finely powdered than the charcoal from pine cones, even after the latter had been pulverized repeatedly in a mortar. It therefore may have had a higher decolorizing value, if it could have been more finely powdered.

On the other hand, the aluminized charcoal prepared from the charcoal from pine cones and aluminum sulphate

had a decolorizing value which exceeded even that of animal charcoal. Whether this charcoal could be used to decolorize solutions in general, without effecting the solution otherwise than destroying its color, was not determined.

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