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Activated Carbon Fabric Prepared by Pyrolysis and Activation of Phenolic Fabric

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Dear Sir:

In a recent article we reported on some experimental activated carbon fabric prepared by pyrolysis and activation of a phenolic fabric [1]. The small sample size of our product precluded an extensive characterization. Now we have successfully prepared activated carbon fabric on a much larger scale. Since the technology of activated carbon textiles is progressing at an accelerated rate, it appears appropriate at this time to report our processing conditions. Selected properties of the product are also reported in this letter. Further details will be included with properties of similar carbon materials in a separate paper. That paper will be concerned with final sorption and textile properties rather than elusive carbonization and activation details, which are usually of a proprietary nature.

An unusual feature of processing in the present work was the use of a production-size high-vacuum furnace. By this method we hoped to rupture the possible closed pores which are sometimes known to be unavailable for sorption. It was hoped also that a highly sorbent carbon could be obtained by pyrolysis steps only. However, a subsequent activation step (with CO_2) at atmospheric pressure was found to be necessary.

A phenolic (Kynol)[1] twill fabric, weighing 6.9 oz/yd^2 was used as the precursor. Initially, the fabric gave a positive iodine test for starch or its derivatives. Therefore, it was desized with a diastatic amylolytic enzyme. The pyrolysis steps described below were then conducted in the large vacuum furnace. Uniform control of temperature and pressure was not possible in the furnace until evolution of gaseous decomposition products ceased.

In the initial pyrolysis step, approximately 3 yd^2 of the desized phenolic fabric weighing 676 g was gradually heated in a nitrogen atmosphere for 250 min while the pressure was slowly reduced until relatively steady conditions of 504°C and 160 microns pressure were finally obtained. These conditions were held for 60 min. Carbonization and degassing in this manner resulted in a 36.5% weight loss from the phenolic fabric. The product sorbed 4% CCl_4 by weight from a saturated vapor at 25°C in a static system.

Next, a further temperature rise (pyrolysis) with degassing was conducted over a 13-h interval until the temperature and pressure reached 910°C and 250 microns, respectively. A rapid pressure drop to 0.5 microns at 910°C indicated termination of degassing. At this point the fabric was assumed to be completely carbonized. The higher temperature pyrolysis produced an 11.1% weight loss based on the above lower temperature pyrolysis product. In this second step the CCl_4 vapor sorption level increased to only 7%.

Carbon activation was conducted on four smaller samples of the above fabric. Each sample weighed approximately 23 g, measured 9 × 16 in., and was placed on a separate perforated Inconel shelf in a 1620-in.^{3} laboratory muffle furnace at atmospheric pressure. The samples were collectively heated to 900°C in a nitrogen atmosphere, activated for 5 min with 5 l./min CO_2 flowing through the furnace, and then allowed to cool while the chamber was quickly purged with nitrogen. The carbon fabric activation results are as follows:

Sample	Activation Wt Loss, %[a]	Weight, oz/yd^2	Surface Area, m^2/g	CCl_4 Vapor Sorption, %
A	37	5.4	—	—
B	27	—	1342	73
C	25	—	1127	59
D	31	—	1385	60

[a] Activation weight losses are not cumulative but are in addition to pyrolysis weight losses at 504°C and 910°C.

Activation with CO_2 produced significant increases in sorption capacity, as indicated by the impressively high surface area and CCl_4 sorption values for Samples B, C, and D. It is anticipated that similar results would have been obtained for Sample A, which was reserved for future textile tests.

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Literature Cited

1. Arons, G. N. and Macnair, R. N., Activated Carbon Fiber and Fabric Achieved by Pyrolysis and Activation of Phenolic Precursors, *Textile Res. J.* **42**, 60–63 (1972).

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[1] Kynol is a product and trademark of the Carborundum Company, Niagara Falls, N. Y. Citation of this and subsequent trade names does not constitute an official endorsement by the US Army Natick Laboratories.