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IMPERVIOUS CARBON ARTICLE AND METHOD OF FORMING THE SAME

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Our invention relates to an impervious article which shall be suitable for use in the handling of acids or organic solvents and to a method of producing the same.

Because of the chemical inertness of carbon, pipes, pumps and reaction vessels formed thereof possess very desirable properties for use in the chemical industry, in the holding of corrosive liquids and solvent reaction mixtures. However, the well known porosity and permeability of all artificial forms of pure carbon material is objectionable. The present invention relates to an improved method of rendering such carbon articles impervious.

It has heretofore been proposed to impregnate carbon articles with various grades of pitch. Such articles withstand dilute acid and salt solutions when cool but at elevated temperatures the pitch tends to ooze out of the carbon in globules, and porosity develops in the pitch causing leaks as well as discoloration of the solutions in contact with the material. Moreover, the pitch is readily attacked by most organic solvents. Various chemical methods of hardening pitch and increasing its resistance to chemicals and heat are known but such treatment of the pitch prior to its use as an impregnating medium renders the pitch so viscous that complete infiltration into large carbon articles becomes impossible.

The present invention avoids these difficulties and yields an impregnated carbon article of greater resistance to the action of both chemicals and heat than any similar previously known product. For example a pitch impregnated concrete material has been disclosed in the prior art which is capable of resisting 80% sulphuric acid but which will rapidly disintegrate in 90% to 96% acid. Impregnated carbon articles made according to the present invention are entirely stable in 90% sulphuric acid and but slowly acted upon in concentrated 96% sulphuric acid. Moreover, such articles may be heated to 250° C. or even higher, without the exudation of pitch from the central portions of the carbon material to the surface.

Our process comprises impregnating the carbon articles with pitch, chemically hardening the pitch to seal it within the pores of the carbon and thermo-hardening of the resins formed in the preceding step. If desired a further step may be included, such step consisting of depositing a protective film on the carbon surface.

The impregnation may take place in any known manner; complete saturation is preferable although not essential. Thus we have found that carbon articles, which take up from 12% to 15% of their weight of pitch for complete saturation, are effectively sealed by additions of 8% to 10% by weight when the pitch is properly resinified. Hard pitch gives a more chemically resistant product after resinification than soft pitch, thus for example the order of maximum chemical stability increases from the pitch known as Barrett No. 15 to the pitch known as Barrett No. 33.

Various chemical reagents may be used in the first resinifying step, such as sulphur vapor, sulphur monochloride, etc. We prefer to treat the impregnated article with 96% concentrated sulphuric acid at a temperature of 175° to 200° C. followed by cooling and removal of the excess acid with water.

The second resinifying step consists in a thermal hardening of the pitch resins formed in the preceding step by heating in air. We prefer to continue this thermal hardening for one to two hours at temperatures of from 200° to 250° C. The time and temperature conditions are not critical so far as effective sealing of the pitch within the carbon is concerned. They are more critical however in respect to the chemical resistance of the pitch sealed carbon to strong sulphuric acids and solvents.

As a method of comparative determination of the chemical stability of articles made in accordance with our invention we place an acid in contact with the impregnated article and let it stand a long time. This acid is afterwards placed in a tintometer and the height of the column necessary to match a standard depth of color is noted. This height is proportional to the chemical stability or resistance of the product. Carbon articles were impregnated with various grades of pitch and treated for one hour at 200° C. with the following results:

Grade of pitch	Tintometer readings
Barrett No. 15	140
Barrett No. 23	325
Barrett No. 33	400

Similar tests were made with impregnated articles subjected to sulphuric acid treatment. The results are as follows:

Temperature	Time	Tintometer readings
°C.	*Minutes*	
150	10	8
175	5	110
175	10	220
175	15	350
175	60	390

Impregnated carbon articles treated with sulphuric acid for fifteen minutes at 175° C. were

given an air bake and tested with the following results:

Temperature	Time	Tintometer readings
°C.	*Hours*	
175	1	300
200	1	390
200	2	400
200	3	340
200	6	290
250	1	510
250	2	420

Articles treated as above described have a particularly desirable characteristic especially when sulphuric acid is used in the first resinifying step. This is that the surface of the carbon is clean on the outside making it possible to apply a further protective film or coating designed to give a further protection to the article against the action of corrosive or highly solvent solutions which would attack even the resinified pitch. Such protective coatings may consist of sodium silicate, aluminum silicate or organic resins such, for example, as the resin formed by the conjoint polymerization of mixtures of vinyl chloride and vinyl acetate.

We claim:

1. The method of forming impervious carbon articles which comprises forming the carbon article, impregnating said article with pitch, resinifying said pitch with concentrated sulphuric acid and subsequently baking said article.

2. Method of forming impervious carbon articles which comprises impregnating the carbon article with pitch, treating the impregnated article with concentrated sulphuric acid at temperatures of about 175° to 250° C., removing the excess acid and thermally hardening the resinified pitch by heating in air at temperatures of from about 200° to about 250° C.

3. Method of rendering a porous article of a substance resistant to organic solvents impervious, which comprises impregnating the article with pitch, treating the impregnated article with concentrated sulphuric acid at temperatures of about 175° to 250° C., removing the excess acid and thermally hardening the resinified pitch by heating in air at temperatures of from about 200° to 250° C.

4. An article formed of a porous body resistant to organic solvents containing a filler identical with that formed by resinified pitch in the presence of concentrated sulphuric acid and thereafter baking said pitch at temperatures of about 200° to 250° C.

5. An article resistant to organic solvents consisting of a porous substance resistant to said solvents, impregnated with a material substantially identical with that formed by resinified pitch in the presence of concentrated sulphuric acid and thereafter baking said pitch in air.

6. Method of rendering pervious articles impervious which comprises impregnating the article with a pitch which is resinified by concentrated sulphuric acid, treating the pitch with concentrated sulphuric acid at elevated temperatures, removing the excess acid and thermally hardening the resinified pitch by baking at temperatures of about 175° to 250° C.

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