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ACTIVATED CARBON TABLET

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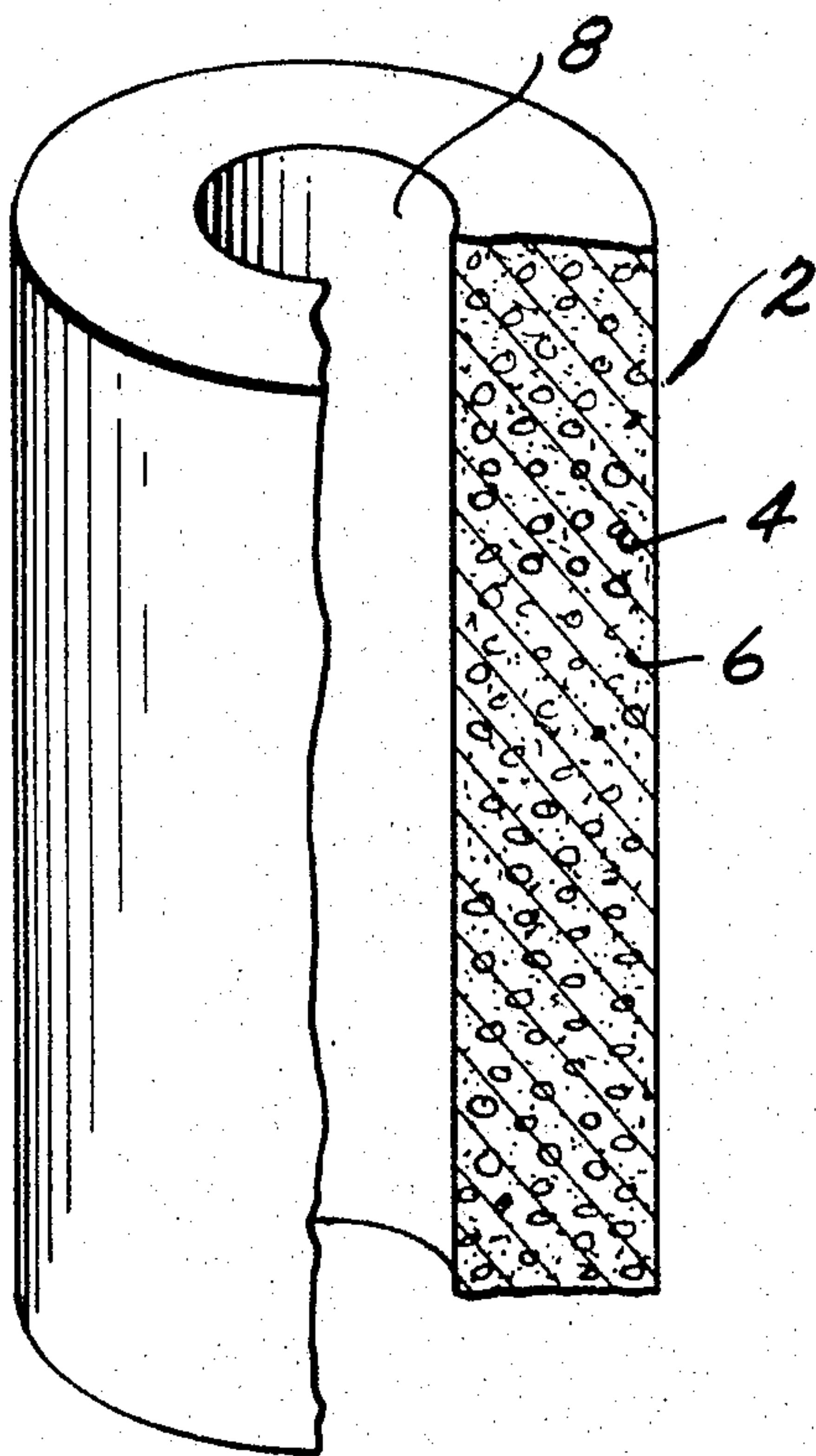


Fig. 1.

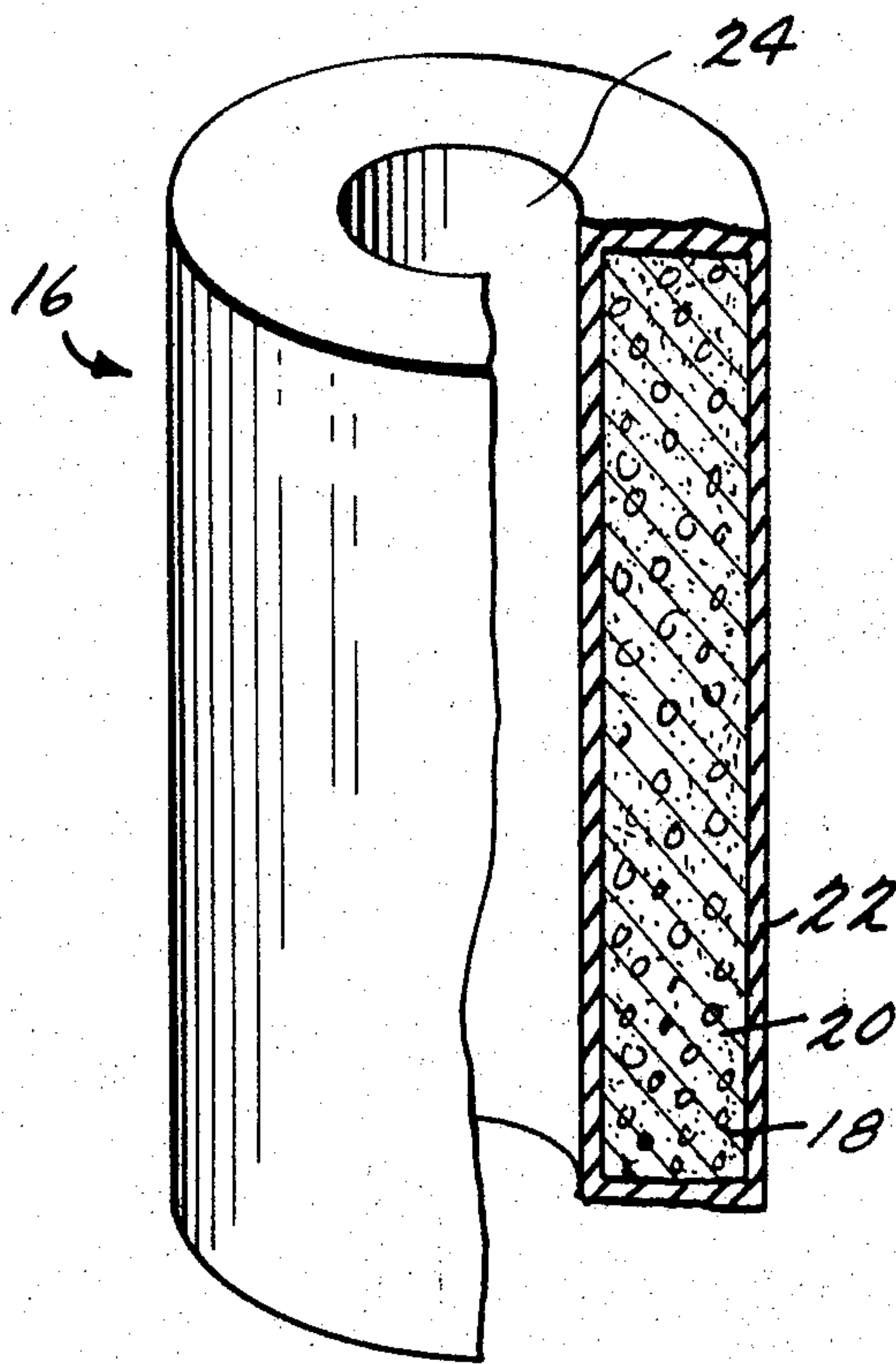


Fig. 3.

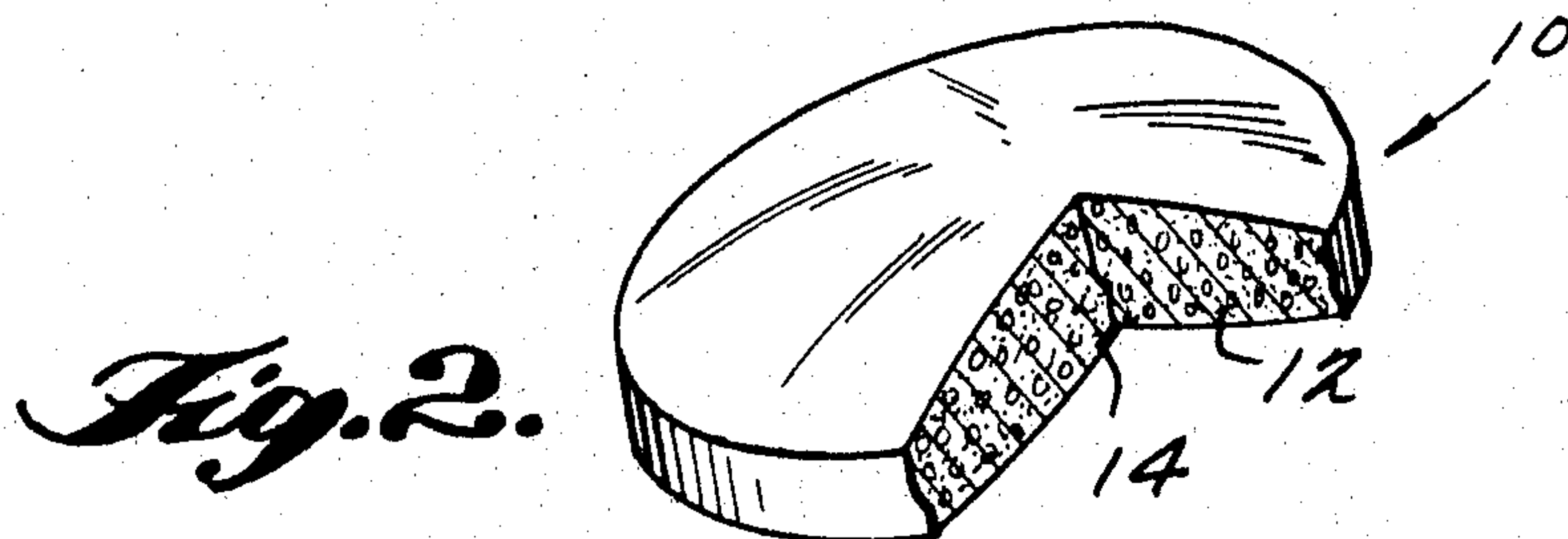


Fig. 2.

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ACTIVATED CARBON TABLET

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8 Claims

The present invention relates to a decolorizing carbon tablet.

In the use of pulverized decolorizing carbons a serious problem exists in plant practice in emptying the containers in which the material is received. Dust permeates the area causing severe plant cleaning problems and may even be an explosion hazard. In many cases where large quantities of carbon are used, separate buildings for emptying the bags are constructed and the large users have built underground storage tanks and the carbon is received in bulk and dropped into these storage tanks in closed chutes. Additionally, the men assigned to placing the carbon in the solution require premium pay due to the unpleasant task. The dirt caused by the carbon requires excess time and soap to wash off.

Powdered activated carbon, more particularly, is used extensively in the decolorizing of liquid materials such as dry cleaning solvents, e.g., perchloroethylene, and hydrocarbon solvents such as Stoddard solvent and naphtha, cane sugar solutions, corn sugar solutions, beet sugar solutions and various organic solutions. As indicated, in the transfer of the carbon to the solution to be treated there is a dust problem.

It is an object of the present invention to reduce the dusting problem occurring with activated carbon.

Another object is to prepare a novel carbon tablet.

A further object is to prepare an activated carbon tablet containing a binder which is readily soluble in dry cleaning solvents.

Still further objects and the entire scope of applicability of the present invention will become apparent from the detailed description given hereinafter; it should be understood, however, that the detailed description and specific examples, while indicating preferred embodiments of the invention, are given by way of illustration only, since various changes and modifications within the spirit and scope of the invention will become apparent to those skilled in the art from this detailed description.

It has now been found that these objects can be attained by forming the divided activated carbon into solid tablets which can be handled easily. The carbon particles are bound together by an aliphatic hydrocarbon detergent. It has been found critical to employ the aliphatic hydrocarbon sulfonic acid salts as the binder since none of the other detergents tested was satisfactory as binders.

The binder should be one which readily dissolves or disperses in the solvent or solution to which the tablet is added so that the powdered or particulate activated carbon becomes available to decolorize the solution. It is also important, therefore, that the binder be one which does not bind the tablet together so tightly that it will not disintegrate in the solution.

The invention is particularly adapted for use with tablets to be added to dry cleaning solvents such as perchloroethylene, as well as petroleum solvents such as Stoddard solvent and naphtha, and fluorochloroalkane dry cleaning solvents such as 1,1,1-trichloro-2,2,2-trifluoroethane. It is also useful for preparing tablets to be added to cane sugar solutions, corn sugar solutions, beet sugar solutions and various organic solvent solutions.

The invention will be understood best in connection with the drawings wherein:

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FIGURE 1 is a view partially broken away of one form of tablet according to the invention;

FIGURE 2 is a similar view of another form of the invention; and

FIGURE 3 is a similar view partially broken away and in section of still another modification.

Referring more specifically to FIGURE 1 of the drawings, there is provided a cylindrical tablet 2 composed of activated carbon particles 4 bound together by a detergent which is the sodium salt of a sulfonated aliphatic hydrocarbon having 10 to 13 carbon atoms as an adhesive designated at 6. In order to accelerate the disintegration of the tablet by exposing a larger surface area, it can have a central opening 8 running therethrough.

FIGURE 2 shows a solid tablet 10 formed of activated carbon particles 12 bonded together by adhesive 14 of the sodium salt of a sulfonated petroleum aliphatic hydrocarbon having 10 to 13 carbon atoms.

To make the tablets more pleasing to the eye, as shown in FIGURE 3, the tablet 16 has a white cover 22 surrounding the activated carbon particles 18 bound by the adhesive 20 of the sodium salt of a sulfonated petroleum aliphatic hydrocarbon sulfonate. The cover is composed of a filter aid such as diatomaceous earth bound together with the same adhesive as that used for the carbon particles. Since a filter aid is normally used in all applications where powdered activated carbon is employed, there is no harm in using this outer jacket.

As the sulfonated aliphatic hydrocarbon salt binder there are normally used soluble salts such as the alkali metal, ammonium and amine salts of sulfonated aliphatic hydrocarbons containing 8 to 18 carbon atoms, preferably 10 to 13 carbon atoms. Thus, there can be used sodium dodecane sulfonate, potassium decane sulfonate, sodium salt of sulfonated petroleum aliphatic hydrocarbons having 8 to 18 carbon atoms, ammonium tridecane sulfonate, potassium salt of sulfonated petroleum aliphatic hydrocarbons having 10 to 13 carbon atoms. The sodium salts are preferred.

While the tablet can be prepared by using the wetting agent alone, it has been found preferable to use this binder in solution in a solvent such as perchloroethylene, 1,1,1-trichloroethane, 1,1,1-trichloro-2,2,2-trifluoroethane, 1,2-dichloro - 1,1,2,2 - tetrafluoroethane, Stoddard solvent, hexane, toluene, benzene, xylene, carbon tetrachloride, chloroform, acetone, methyl ethyl ketone, methyl chloroform, 2,2-dichloropropane, 1,2,3-trichloropropane, dichloroethylene, etc. Preferably, the solvent employed with the binder is the same solvent used in the dry cleaning solution to avoid the introduction of extraneous matter. The preferred solvents are hydrocarbons and halo-hydrocarbons.

The activated carbon tablets which are formed with binders that contain solvents gradually lose the solvent due to evaporation but still hold together. There was noted an improvement in strength while the solvent partially evaporated, but the strength leveled off before all the solvent was removed. Preferably, a solvent is used together with the binder. The weight loss of the tablets due to solvent evaporation was not significant.

The tablets preferably contain 30 to 50% of the aliphatic hydrocarbon petroleum sulfonate binder and 70 to 50% of the activated carbon. Below about 30% of binder the tablet does not hold together too well, and above 50% of binder the tablet tends to become sticky. However, with some sacrifice in properties the binder can be dropped to as low as 20% or 25%, or increased to as much as 55% or 60%.

When a solvent is employed, the binder is 30 to 100% of the total of binder and solvent. Usually, 15 to 50% of the mixture of binder and solvent is solvent.

Modifying agents can be added to the petroleum sulfonate binder, but they are not essential. Preferably, at least 50% of the binder formulation (excluding solvent) is the petroleum sulfonate. Examples of modifying agents which can be used in minor amounts are alkali metal diphenyl ether sulfonates having an 8 to 18 carbon atom side chain, alkylphenol-ethylene oxide condensates having 8 to 18 carbon atoms in the alkyl group and 4 to 20 ethylene oxide units in the molecule, polyethylene glycol, e.g., of molecular weight 20,000, and polypropylene glycol, glyceryl esters of higher fatty acids, e.g., glyceryl monooleate, glyceryl monostearate an glyceryl monopalmitate. There can even be employed water as either a diluent or solvent, although normally water is not a preferred solvent according to the invention.

The tablets can be spherical, cylindrical, rectangular, elliptical or other shape.

The tablets can vary in weight from about 0.002 to 1 pound. In making a 1/2 pound cylindrical table it will have a diameter of 2 1/2 inches and height of 4 inches with a 1/2 inch diameter hole running therethrough. A 1/2 pound tablet would be proportionately larger in size. On the other hand, tablets having a diameter of 10 mm. and a height of 2 mm. also can be formed according to the invention.

The tablets are formed in conventional fashion in a press or briquetting machine. The pressure can be as low as that required to close the press up to 20,000 p.s.i. With finely divided activated carbon the pressures employed to form the tablet are usually from 1,800 or 2,000 p.s.i. up to 20,000 p.s.i. With granular activated carbon, e.g., 8 x 30 mesh (carbon which pass an 8 mesh sieve but is retained on a 30 mesh sieve, U.S. Sieve Series), the pressure is desirably not over 5,000 p.s.i. With such large granules pressures above 600 p.s.i. tended to crush the granules and, hence, are not preferred.

The activated carbon employed can be as fine as 325 mesh. The powdered carbon employed usually had at least 72% which passed a 325 mesh sieve (U.S. Sieve Series). As used in the present specification, powdered carbon is carbon particles up to 100 mesh, while granular carbon is carbon particles having a size larger than 100 mesh.

The activated carbon employed can have an iodine number of from 400 to 1,200, but for best results has an iodine number of 900 to 1,100. The surface area also is preferably at least 900 m.²/g. and can be as much as 1,400 m.²/g. The molasses number is preferably at least 195 and can be 400 or more. Typical examples of preferred activated carbons are Pittsburgh BL and Pittsburgh SGL which have the following properties:

Property	BL	SGL
Total surface area, m. /g.	1,000-1,100	950-1,050
Apparent density, g./cc.	0.51	0.48
Real density, g./cc.	2.1	2.1
Pore volume (within particle) cc./g.	0.90	0.85
Iodine number, minimum, mg./g.	1,000	900
Molasses number, minimum	230	200
Ash, maximum, weight percent	8.5	8
Screen analysis, minus 325 U.S. mesh, percent	65-75	

Pittsburgh SGL passes an 8 mesh screen but the vast majority of it does not pass a 30 mesh screen.

Less preferably there can be used other activated carbons such as Darco Premium or Nuchar CEE-N.

The tablets or briquets made according to the present invention can be added to the dry cleaning solution, sugar solution, corn sugar solution or beet sugar solution at any appropriate point. For example, they can be added to the button trap or any other convenient point in the cleaning cycle.

Unless otherwise indicated, all parts and percentages are by weight.

In the following examples Formulation A had the following composition:

	Percent
Sodium salt of a sulphonated petroleum aliphatic hydrocarbon of 10 to 13 carbon atoms	65
Sodium salt of alkylated (C ₈ -C ₁₈) diphenylether sulfonate	1
Alkyl (C ₈ -C ₁₃) phenol-ethylene oxide condensate (9 ethylene oxide units)	5
Polyethylene glycol, mol. wt. 20,000	2
Hexylene glycol	2
Glyceryl monooleate	5
Perchloroethylene	17
Water	3

Examples 2 and 5 illustrate the unsatisfactory results obtained when using detergents other than the aliphatic hydrocarbon sulfonates. In addition, in other tests satisfactory briquets could not be prepared from the activated carbon and the following detergents: sodium alkylbenzene sulfonate, sodium dioctyl sulfosuccinate, sorbitan monooleate, sorbitan tristearate, bis (tridecyl) ester of sodium sulfosuccinic acid.

EXAMPLE 1

75 grams of Formulation A and 120 grams of Pittsburgh BL activated carbon were placed in a Cincinnati Mix Muller for 15 minutes.

Briquets were made in a Carver press (1 1/4 inch ram) at 20,000 pounds with a 10 second hold.

The briquets were allowed to stand in the air overnight and were fairly hard and were satisfactory for use. They did not crumble on standing or in handling, but readily disintegrated when placed in perchloroethylene.

EXAMPLE 2

When 100 grams of another detergent mix of 40% ethoxylated nonyl phenol, 5% diisopropyl amine salt of alkylbenzene sulfonic acid and 55% mineral oil, was milled with 132 grams of a similar Pittsburgh activated carbon to that used in Example 1 and then briquetted in the same manner, the air dried briquets crumbled when pinched in the fingers and, hence, were unsuitable. The same result was obtained when using 40 grams of the detergent mix to 60 grams of the activated carbon and pressing for 15 minutes at (a) 8,000 p.s.i. or (b) 4,000 p.s.i. In each case the briquet was very soft and crumbly.

EXAMPLE 3

The procedure of Example 1 was repeated using 150 grams of Formulation A and 240 grams of the same activated carbon and using briquetting pressures of (a) 16,000 p.s.i. and (b) 8,000 p.s.i. and allowing the briquet to air dry. A briquet made at 16,000 p.s.i. which weighed 23.8888 grams lost 2.69% in weight over a period of two weeks and had a total loss in weight of 6.86% over a period of 18 months. The briquets, however, held together well but readily disintegrated when added to perchloroethylene.

A briquet made at 8,000 p.s.i. lost 8.02% in weight over a period of 18 months but was also firm and an acceptable briquet.

EXAMPLE 4

The procedure of Example 3 was used to form briquets from 150 grams of Formulation A and 240 grams of the same activated carbon using briquetting pressures of (a) 4,000 p.s.i. and (b) 2,000 p.s.i. In each case the briquets showed the same good hardness as obtained at 8,000 p.s.i. and 16,000 p.s.i.

EXAMPLE 5

When 60 grams of the same activated carbon as that of Example 1 was employed with 40 to 50 grams of various commercial detergents which were not aliphatic hydrocarbon sulfonates, the briquets obtained were crumbly and unsatisfactory in each case. Specific examples of the

commercial detergents used with the 60 grams of carbon were:

	Grams
(a) Pace Perc -----	48
(b) Staticol -----	40
(c) Plaid -----	47
(d) Perclite -----	49
(e) Paa Maker -----	44
(f) Par 4 -----	49
(g) Tex N -----	50
(h) Merasol -----	46

EXAMPLE 6

Satisfactory briquets were made from (a) 60 grams Pittsburgh BL activated carbon and 40 grams of Formulation A, as well as from (b) 60 grams of the carbon and 54 grams of Formulation A.

EXAMPLE 7

A satisfactory briquet was made from a mixture of 5 grams of Pittsburgh BL carbon and 5 grams of Floklear (an aliphatic petroleum sulfonate detergent made by Pennsalt) using a pressure of 8,000 p.s.i. The briquet was 29 mm. in diameter and 15 mm. high and was firm and strong.

Replacing the BL carbon by the same amount of (a) Darco DC carbon and (b) Darco S-51 carbon gave briquets which were firm but not as strong as those from the BL carbon.

EXAMPLE 8

Briquets made from 60 parts of an activated carbon very similar to Pittsburgh BL and 30 parts of Floklear were used to decolorize and clean dirty perchloroethylene from a dry cleaning establishment. There was obtained considerable improvement as shown in optical density measurements at 450 mμ and 750 mμ. There was added to 100 ml. of the dirty perchloroethylene the indicated amount of the tablets and the mixture was agitated for 3 hours at room temperature, filtered and the optical density measured at 10 cm. depth.

TABLE I

	O.D. at 450 mμ	O.D. at 750 mμ
Blank -----	0.920	0.033
0.5 gram tablet -----	0.703	0.168
1.0 gram tablet -----	0.610	0.125
2.0 gram tablet -----	0.514	0.104
4.0 gram tablet -----	0.520	0.117

At 450 mμ the amount of color is measured and a decrease in value indicates a decrease in color. At 750 mμ turbidity is measured, and an increase in value indicates an increase in turbidity.

EXAMPLE 9

150 grams of Pittsburgh SGL activated carbon (8 x 30 mesh) was stirred with 90 grams of Formulation A. Portions of this mix were placed in the press and subjected to pressures of (a) 0, (b) 600 p.s.i., (c) 1,000 p.s.i., (d) 2,000 p.s.i., (e) 3,000 p.s.i., (f) 4,000 p.s.i. and (g) 5,000 p.s.i. All made tablets which could be handled at once. They were even better after standing for two days. While all of them were satisfactory briquets, (c) and (d) were the best. All of them disintegrated very readily when put into perchloroethylene.

While the briquets prepared in Example 9 were useful, they were not as good as those made from the Pittsburgh BL, a powdered activated carbon.

EXAMPLE 10

A series of tablets were made using a Pittsburgh activated carbon substantially the same as BL and using the indicated amounts of Component A (Petrosul 744-LC, a sodium petroleum sulfonate) and Component B (perchloroethylene). One tablet of each type was prepared at

2,000 p.s.i. and at 10,000 p.s.i. with the results shown in Table II.

TABLE II

Component A (grams)	Component B (grams)	Carbon (grams)	Results
10 -----	90	90	Neither tablet held together
20 -----	80	90	Do.
30 -----	70	90	Both were all right but slightly soft.
40 -----	60	90	Both were all right.
50 -----	50	90	Do.
100 -----	0	90	Do.
80 -----	20	90	Do.
60 -----	40	90	Do.

EXAMPLE 11

Good tablets were made by making a paste of 50 grams Petrosul 744-LC and 50 grams of water, mixing with 90 grams of the activated carbon of Example 10 and pressing at (a) 2,000 p.s.i. and (b) at 10,000 p.s.i. They disintegrated when placed in perchloroethylene. The tablets were not as good as those where perchloroethylene was used rather than water.

EXAMPLE 12

Tablets were prepared using 50 grams of Petrosul 744-LC with 50 grams of various solvents. After mixing the Petrosul and solvent there were added 90 grams of the same activated carbon as in Example 10 and tablets made at (a) 2,000 p.s.i. and (b) 10,000 p.s.i.

The solvents used were:

- (1) 1,1,1-trichloroethane
- (2) Acetone
- (3) Methyl alcohol
- (4) Carbon tetrachloride
- (5) Stoddard solvent
- (6) Hexane
- (7) Toluene

The results were as follows:

- (1) Made good tablets at both pressures which disintegrated well in perchloroethylene.
 - (2) The high pressure made fair tablets, but the low pressure was unsatisfactory.
 - (3) Neither tablet held together.
 - (4) Both tablets were good and redispersed well in perchloroethylene.
 - (5) Both tablets were fair, but could be used.
 - (6) Both tablets were good in all respects.
 - (7) Both tablets were good in all respects.
- The chlorinated solvents were the best.

EXAMPLE 13

A satisfactory tablet was made by using 60 parts of Pittsburgh BL activated carbon with 40 parts of a mixture of sodium aliphatic petroleum hydrocarbon sulfonate (having 10 to 13 carbon atoms) and perchloroethylene when the mixture contained:

- (a) 65% sulfonate-35% perchloroethylene
- (b) 83% sulfonate-17% perchloroethylene
- (c) 70% sulfonate-30% perchloroethylene.

The tablets were made at 2,000 p.s.i.

EXAMPLE 14

Satisfactory briquets were made from a mix of (a) 15 grams Hyflo Super Cel filter aid and 35 grams Pittsburgh PSC carbon and 45 grams Formulation A and (b) 25 grams Hyflo Super Cel filter aid and 25 grams Pittsburgh PSC carbon and 45 grams Formulation A. Two satisfactory briquets were made using briquetting pressures of (a) 2,000 p.s.i. and (b) 10,000 p.s.i.

The tablets of the present invention are characterized by being substantially dust free and they are resistant to crumbling upon handling. However, they disintegrate readily when placed in dry cleaning solvents.

We claim:

- 1. A tablet consisting essentially of 40 to 80% acti-

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vated carbon and 60 to 20% of an 8 to 18 carbon atom water soluble aliphatic hydrocarbon sulfonate detergent as a binder therefor.

2. A tablet according to claim 1 wherein the carbon is 50 to 70% of the total tablet.

3. A tablet according to claim 1, including a minor amount of solvent, the binder being between 30 and 100% of the total of binder and solvent, the solvent being selected from the group consisting of hydrocarbons and chlorinated hydrocarbons.

4. A tablet according to claim 3 wherein the solvent is a chlorinated hydrocarbon.

5. A tablet according to claim 4 wherein the solvent is perchloroethylene.

6. A tablet according to claim 1 wherein the sulfonate is a sodium sulfonate and there are 8 to 18 carbon atoms in the hydrocarbon group.

7. A tablet according to claim 6 wherein the activated carbon is one having an iodine number of 900-1,100.

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8. A tablet according to claim 7 wherein the activated carbon has a particle size of not over 100 mesh.

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