US005914294A

United States Patent [19]

Park et al.

[11] **Patent Number: 5,914,294**

[45] **Date of Patent: Jun. 22, 1999**

[54] **ADSORPTIVE MONOLITH INCLUDING ACTIVATED CARBON AND METHOD FOR MAKING SAID MONLITH**

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[21] Appl. No.: **08/636,700**

[22] Filed: **Apr. 23, 1996**

[51] **Int. Cl.**[6] **B01J 20/02**; B01J 21/18; C04B 33/24

[52] **U.S. Cl.** **502/417**; 502/427; 502/436; 502/180; 501/100; 501/143

[58] **Field of Search** 502/417, 427, 502/436, 180; 501/100, 143

[56] **References Cited**

U.S. PATENT DOCUMENTS

Re. 25,400	6/1963	Doying	252/428
617,079	1/1899	Catlett .	
1,524,843	2/1925	Ruprecht .	
1,589,081	6/1926	Adler	502/80
1,985,840	12/1934	Sadtier	131/31
2,108,860	2/1938	Kauffman	131/31
2,391,312	12/1945	Ewing et al.	252/235
2,439,358	4/1948	Divoll	74/5
2,439,538	4/1948	Burgess	252/265
2,455,509	12/1948	Luaces	18/55
2,951,087	8/1960	Hauser	260/448
3,089,195	5/1963	Woodburn, Jr.	18/47.5
3,454,502	7/1969	Hiltgen et al.	252/428
3,592,779	7/1971	Kiikka	252/421
3,632,385	1/1972	Schmitt	117/46
3,690,634	9/1972	Enya	266/33 R
3,825,460	7/1974	Yoshikiawa et al.	156/296
3,859,421	1/1975	Hucke	423/445
3,864,277	2/1975	Kovach	252/423
3,891,574	6/1975	Kobayashi et al.	252/421
3,927,186	12/1975	Vinton et al.	423/447
3,960,761	6/1976	Bürger et al.	252/421
3,960,771	6/1976	Tanaka et al.	252/446
4,029,600	6/1977	Schmitt, Jr. et al.	252/444
4,051,098	9/1977	Takemura et al.	260/38
4,058,483	11/1977	Henbest	252/446
4,124,529	11/1978	Jüntgen et al.	252/421
4,220,553	9/1980	Krause	252/428
4,225,569	9/1980	Matsui et al.	423/445
4,259,299	3/1981	Hagiwara et al.	423/210
4,338,106	7/1982	Mizuno et al.	55/316
4,399,052	8/1983	Sugino	252/421
4,518,704	5/1985	Okabayashi et al.	502/80
4,677,086	6/1987	McCue et al.	502/62
4,808,559	2/1989	Sommer et al.	502/63
4,923,843	5/1990	Saforo et al.	502/415
4,954,469	9/1990	Robinson	502/80
4,968,651	11/1990	Crabtree	502/63
4,999,330	3/1991	Bose et al.	502/402
5,043,310	8/1991	Takeuchi et al.	502/404
5,215,690	6/1993	Golino et al.	264/29.4
5,306,675	4/1994	Wu et al.	502/5
5,356,852	10/1994	DeLiso et al.	502/402
5,376,609	12/1994	Guile	502/62
5,389,325	2/1995	Bookbinder et al.	264/177.12
5,403,809	4/1995	Miller et al.	502/416

FOREIGN PATENT DOCUMENTS

1567491	4/1969	Germany .
59-69146	4/1984	Japan .
2409	9/1865	United Kingdom .
3078	9/1865	United Kingdom .
341233	1/1931	United Kingdom .

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Assistant Examiner—In Suk Bullock
Attorney, Agent, or Firm—Jones & Askew, LLP

[57] **ABSTRACT**

An adsorptive monolith made by extruding a mixture of activated carbon, a ceramic forming material, a flux material, and water, drying the extruded monolith, and firing the dried monolith at a temperature and for a time period sufficient to react the ceramic material together and form a ceramic matrix. The extrudable mixture may also comprise a wet binder. The monolith has a shape with at least one passage therethrough and desirably has a plurality of passages therethrough to form a honeycomb. The monolith may be dried by vacuum drying, freeze drying, or control humidity drying. The monolith is useful for removing volatile organic compounds and other chemical agents such as ozone from fluid streams. Particularly useful applications include adsorptive filters for removing ozone from xerographic devices and other appropriate office machines and volatile organic compounds from automobile engine air intake systems.

63 Claims, 3 Drawing Sheets

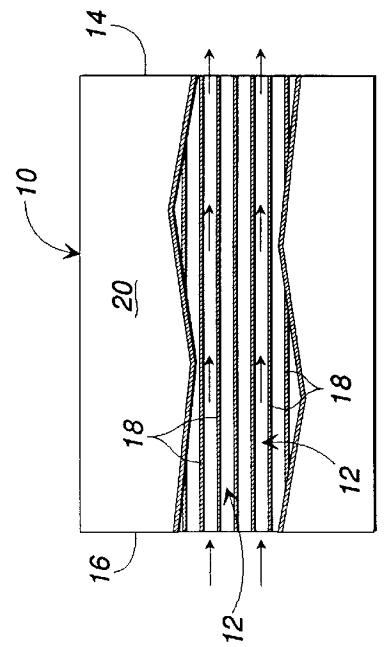

FIG. 2

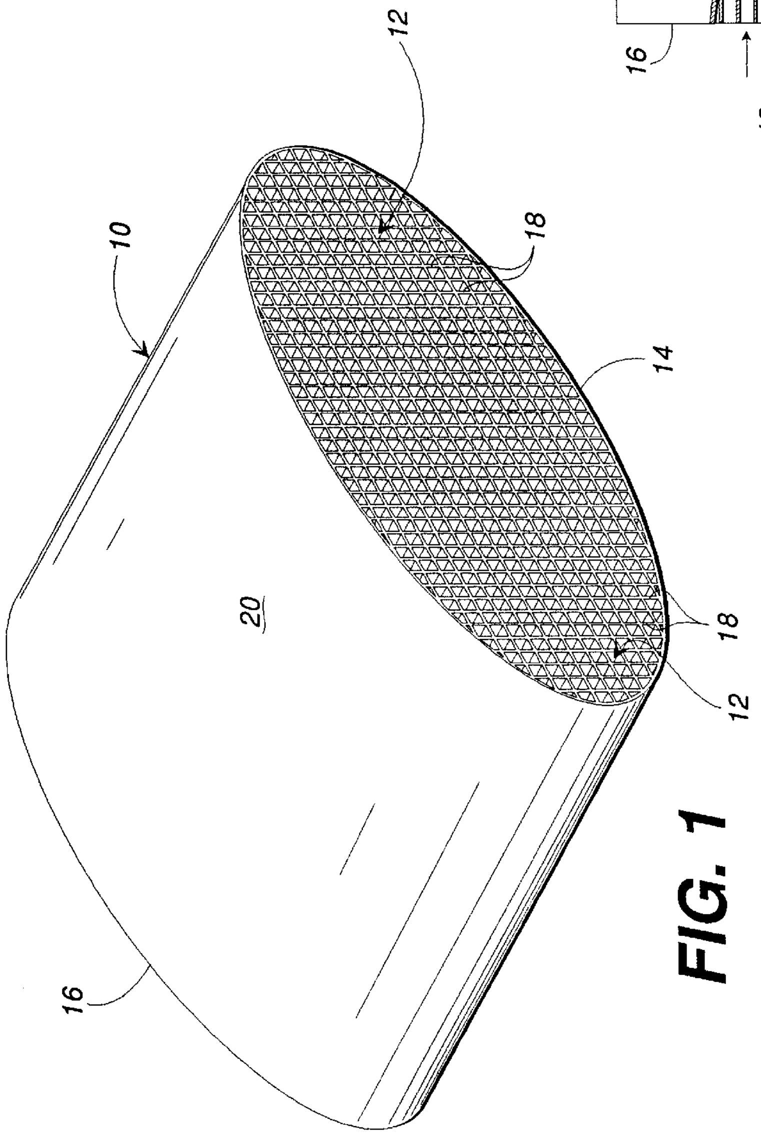

FIG. 1

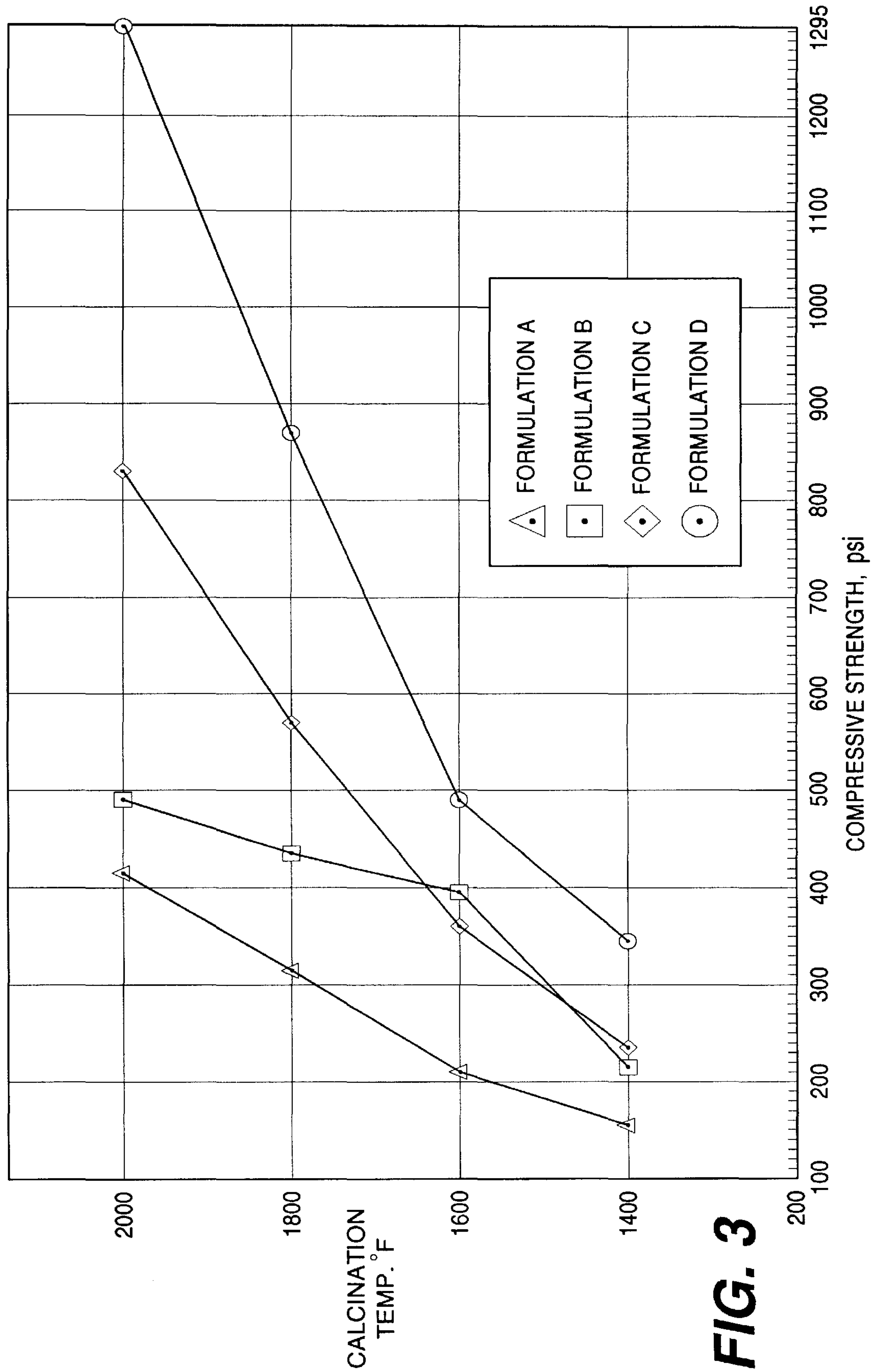
CALCINATION TEMP. °F
2000
1800
1600
1400
200
COMPRESSIVE STRENGTH, psi
100
200
300
400
500
600
700
800
900
1000
1100
1200
1295
FORMULATION A
FORMULATION B
FORMULATION C
FORMULATION D

FIG. 3

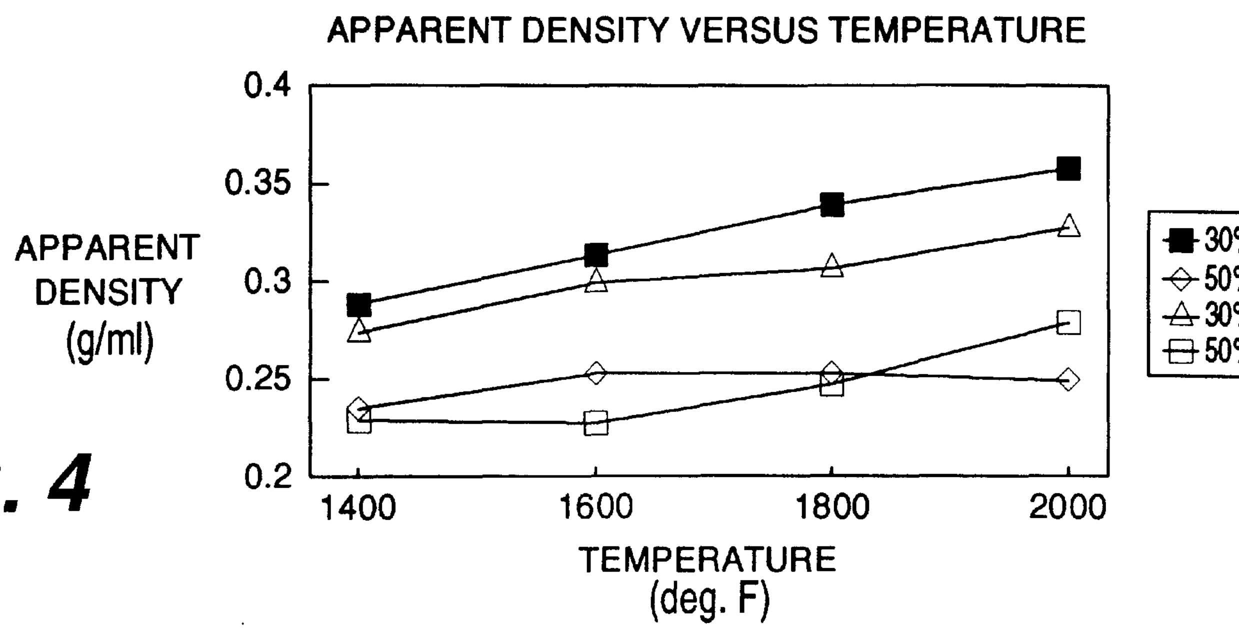
APPARENT DENSITY VERSUS TEMPERATURE
APPARENT DENSITY (g/ml)
0.4
0.35
0.3
0.25
0.2
1400
1600
1800
2000
TEMPERATURE (deg. F)

FIG. 4

ADSORPTIVE MONOLITH INCLUDING ACTIVATED CARBON AND METHOD FOR MAKING SAID MONLITH

TECHNICAL FIELD

This invention relates to adsorptive monoliths including activated carbon and more particularly to adsorptive monoliths including ceramic material and activated carbon and using said monolith to remove volatile organic compounds, ozone, and other chemical agents from fluid streams.

BACKGROUND OF THE INVENTION

Activated carbon is useful in the removal of chemical agents such as volatile organic compounds from fluid streams and is also useful as a catalyst substrate for special applications. To remove chemical agents from a fluid stream with activated carbon, the fluid stream is directed adjacent the activated carbon. The activated carbon can be in the form of particles in a packed column, a coating on a substrate, a monolith with passages for fluid flow therethrough, and the like.

It is desirable in some activated carbon applications to have a high rate of fluid flow adjacent to the activated carbon and a low level of back pressure. Thus, packed columns of activated carbon are sometimes unsuitable because of the high level of back pressure created. Formed bodies containing activated carbon and having open passages therethrough, such as a honeycomb-shaped activated carbon monolith, are desirable for applications wherein a reasonably high rate of fluid flow and a low level of back pressure are required, but formation of such shapes with a level of strength sufficient to withstand handling and use as an adsorbent filter is problematic. Activated carbon monoliths formed without a binder do not have sufficient strength for some applications.

U.S. Pat. No. 4,518,704 to Okabayashi et al. discloses a formed body comprising activated carbon and a ceramic material. This structure has improved strength properties but Okabayashi teaches firing at a temperature of 1100° C. for a period from 1 to 4 hours to achieve desired bonding and strength. Firing at such a high temperature and for such a long period of time is economically undesirable.

Another problem with making adsorptive monoliths comprising activated carbon and a ceramic material is that it is difficult to extrude a mixture of activated carbon and ceramic forming material without a high level of water in the mixture due to the high porosity of the activated carbon. To successfully extrude a mixture of activated carbon and ceramic forming material into a shape such as a honeycomb, a water content of 30 to 65 percent by weight is required. This moisture must be substantially removed from the extruded monolith before firing to protect the integrity of the formed monolith. A ceramic article subjected to increased temperature during firing, without first having been relieved of most of its moisture content, will usually suffer significant damage in the forms of cracks, pop-outs or explosions due to rapid conversions of its remaining moisture to steam.

Drying of a wet, extruded monolith of ceramic forming material and activated carbon is a sensitive process. An unfired ceramic product generally shrinks as it loses moisture, and a monolith can crack if the rate of moisture loss from the monolith during drying is not uniform throughout the monolith.

Accordingly, there is a need for a formed body comprising activated carbon that can be formed by extrusion, can be dried and fired without cracking, can be fired at more

economical conditions such as a lower temperature and a shorter time, has sufficient strength to withstand handling and use as an adsorptive filter, and has a shape which accommodates sufficient fluid flow throughput.

SUMMARY OF THE INVENTION

This invention solves the above-described problems by providing a method of forming an adsorptive monolith comprising extruding an extrudable mixture including an activated carbon, a ceramic forming material, water, and a flux material. The flux material enhances the fusing of the ceramic forming material upon firing by lowering the temperature at which the ceramic forming material fuses and forms ceramic bonds. This allows the monolith to be fired at a lower temperature and for a shorter time. In addition, the invention encompasses methods of drying the wet extruded monolith including vacuum drying, freeze drying, and humidity control drying. Such drying methods allow the wet extruded monolith to be dried without cracking of the monolith.

More particularly, this invention encompasses a method of forming an adsorptive monolith comprising the steps of (a) extruding an extrudable mixture through an extrusion die such that a monolith is formed having a shape wherein the monolith has at least one passage therethrough and the extrudable mixture comprises activated carbon, a ceramic forming material, a flux material, and water, (b) drying the extruded monolith, and (c) firing the dried monolith at a temperature and for a time period sufficient to react the ceramic forming material together and form a ceramic matrix. The extrudable mixture is capable of maintaining the shape of the monolith after extrusion and during drying of the monolith.

A suitable ceramic forming material is ball clay. In addition, the ceramic forming material desirably includes a filler for reducing shrinkage of the monolith during the steps of drying and firing. A suitable filler is calcined kaolin clay.

A suitable flux material is a feldspathic material, particularly, nepheline syenite.

Desirably, the extrudable mixture includes a wet binder for enhancing strength and maintaining the shape of the wet extruded monolith. A particularly suitable wet binder is methylcellulose. Acrylic binders are also suitable and can be used in combination with methylcellulose.

The extrudable mixture can also include sodium silicate which, as a binder, enhances the strength of the monolith during drying and, as a flux material, enhances the strength of the monolith after firing.

Desirably, the adsorptive monolith has a plurality of passages therethrough and is in the shape of a honeycomb.

The extruded monolith may be dried by vacuum drying which includes placing the extruded monolith in a vacuum chamber initially having room ambient temperature and atmospheric pressure within the vacuum chamber, reducing the pressure within the vacuum chamber at a rate and to a level sufficient to freeze the water in the monolith, and maintaining the reduced pressure within the vacuum chamber for a time sufficient for the frozen water to sublime until the monolith is dried. More particularly, the pressure within the vacuum chamber may be reduced, within about 1 minute, from atmospheric pressure to a pressure less than about 1 torr, and desirably within the range from 30 microns to 1 torr.

The method of freeze drying the wet extruded monolith comprises the steps of (1) freezing the water in the extruded

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