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TITLE:

"HIGH SURFACE AREA CARBON BLACKS"

This invention relates to furnace blacks having many important applications, such as fillers, reinforcing agents, pigments and the like. More particularly, the invention relates to furnace blacks having high surface areas.

According to the present invention there is provided a furnace-type carbon black product characterised by having an iodine surface area of at least $600\text{m}^2/\text{g}$, a pH value of less than 5 and a DBP value of at least 160cc/100g.

Preferably the novel blacks are characterised by having an iodine surface area ranging from $800\text{m}^2/\text{g}$ and higher. Also, the preferred carbon blacks have a pH ranging from 2 to 4 with the most preferred being in the range of 3 to 4. With respect to structure level, the preferred DBP values are from 180 to 350, and higher, with the most preferred ranging from 180 to 275 cc/100g.

The novel blacks of the present invention may be obtained by the process described and claimed in our copending application for Letters Patent No. 8023333.

In the process for producing high surface area blacks, the following operation is observed. A

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carbon black-yielding liquid hydrocarbon feedstock is injected substantially transversely into a pre-formed stream of hot combustion gases flowing in a downstream direction at an average linear velocity of at least 500 feet per second. The feedstock is injected transversely in the form of coherent jets into the combustion gases from the periphery of the stream to a degree sufficient to achieve penetration and thereby avoid coke formation on the walls of the carbon forming zone of the reactor. In this instance, however, the feedstock will have been injected into a preformed stream of gaseous combustion products containing added water vapor thoroughly mixed therewith. The amount of added water, as stated earlier, is critical to the successful operation of the present process, and this feature together with the other indicated operations, including requisite overall percent combustion ranges and definite residence times are directly related to the production of the unusually high surface area furnace blacks.

In the preparation of the hot combustion gases employed in preparing the blacks of the present invention, there are reacted in a suitable combustion chamber a liquid or gaseous fuel and a suitable oxidant stream such as air, oxygen, mixtures of air and oxygen or the like. Among the fuels suitable for use in reacting with the oxidant stream in the combustion chamber to generate the hot combustion gases are included any of the readily combustible gas, vapor or liquid streams such as hydrogen, carbon monoxide, methane, acetylene, alcohols, kerosene. It is generally preferred, however, to utilize fuels having a high content of carbon-containing components and, in particular, hydrocarbons. For example, streams rich in methane such as natural

gas and modified or enriched natural gas are excellent fuels as well as other streams containing high amounts of hydrocarbons such as various hydrocarbon gases and liquids and refinery by-products including ethane, propane butane, and pentane fractions, fuel oils and the like. As referred to herein, the primary combustion represents the amount of oxidant used in the first stage of the modular process relative to the amount of oxidant theoretically required for the complete combustion of the first stage hydrocarbon to form carbon dioxide and water. In the present process, the primary combustion may range from about 85 to about 300 percent combustion, with the preferred primary or first stage combustion ranging from 85 to 150 percent combustion. In this manner there is generated a stream of hot combustion gases flowing at a high linear velocity. It has furthermore been found that a pressure differential between the combustion chamber and the reaction chamber of at least 1.0 p.s.i. (6.9 kPa), and preferably of 1.5 (10.3 kPa) to 10 (69 kPa) p.s.i., is desirable. Under these conditions, there is produced a stream of gaseous combustion products possessing sufficient energy to convert a carbon black-yielding liquid hydrocarbonaceous feedstock to the desired carbon black products. The resultant combustion gas stream emanating from the primary combustion zone attains a temperature of at least 2400°F (1316°C), with the most preferable temperatures being at least above 3000°F (1649°C). The hot combustion gases are propelled in a downstream direction at a high linear velocity which is accelerated by introducing the combustion gases into an enclosed transition stage of smaller diameter which may, if desired, be tapered or restricted such as by means of a conventional venturi throat. It is at this point in the process, which is regarded as the

second stage, where the feedstock is forcefully injected into the stream of hot combustion gases.

More particularly, in the second stage where the combustion gases are traveling at high velocity and there exists a gas kinetic head of at least above 1.0 p.s.i (6.9 kPa), a suitable liquid carbon black-yielding hydrocarbon feedstock is injected into the combustion gases, under sufficient pressure to achieved desired penetration thereby insuring a high rate of mixing and shearing of the hot combustion gases and the liquid hydrocarbon feedstock. As a result of this environment, the liquid hydrocarbon feedstock is rapidly decomposed and converted to carbon black in high yields. Suitable for use herein as hydrocarbon feedstocks which are readily volatilizable under the conditions of the reaction are unsaturated hydrocarbons such as acetylene; olefins such as ethylene, propylene butylene; aromatics such as benzene, toluene and xylene; certain saturated hydrocarbons; and volatilized hydrocarbons such as kerosenes, naphthalenes, terpenes, ethylene tars, aromatic cycle stocks and the like. The liquid feedstock is injected substantially transversely from the outer or inner periphery, or both, of the stream of hot combustion gases in the form of a plurality of small coherent jets which penetrate well into the interior regions or core of the stream of combustion gases but not to a depth such that opposing jets would impinge. In practicing this invention, the hydrocarbon feedstock may readily be introduced as coherent streams of liquid by forcing the liquid feedstock through a plurality of orifices having a diameter ranging from 0.01 (0.25 mm) to 0.15 (3.81 mm) inch, and preferably ranging from 0.02 (0.51 mm) to 0.06 (1.52 mm) inch under an injection pressure sufficient to achieve the desired penetration.

The amount of feedstock utilized and/or the amounts of fuel and/or oxidant employed herein will be adjusted so as to result in an overall percent combustion of from 40 to about 60 percent and preferably from about 46 to 57 percent. The overall combustion represents the total amount of oxygen used in the carbon forming process relative to the amount of oxygen required for the complete combustion of the total amount of hydrocarbon used in the carbon forming process so as to yield carbon dioxide and water. The overall combustion is usually expressed as a percentage.

The third stage of the modular process involves the provision of a reaction zone which will permit sufficient residence time for the carbon black forming reaction to occur prior to termination of the reaction by quenching. In general, although the residence time in each instance depends upon the particular conditions and the particular black desired, the residence times of the present process are at least 0.5 second, and preferably at least 1.0 second. Accordingly, once the carbon black forming reaction has proceeded for the desired period of time, the reaction is terminated by spraying thereon a quench liquid, such as water, using at least one set of spray nozzles. The hot effluent gases containing the carbon black products suspended therein are then passed downstream where the steps of cooling, separating and collecting the carbon black are carried out in conventional manner. For example, the separation of the carbon black from the gas stream is readily accomplished by conventional means such as a precipitator, cyclone separator, bag filter, or combinations thereof.

As mentioned hereinbefore, the practice of the above-described process will result in the production of furnace blacks

having high surface areas and excellent conductivity properties when water, in the form of vapor, is included as an essential operation of the process. More particularly, it is not merely the addition of water to the process that results in the production of the high surface areas of the blacks, but rather, the manner of introducing the water, the amount of water and the form in which the water is introduced. All of these features are necessary for the proper operation of the present process. In more detail, the added water, which may be in any physical form at the time of addition, must, however, be in the form of water vapor within the stream of gaseous combustion products prior to the introduction of the liquid feedstock. Furthermore, the added water must be introduced under conditions such that the water is well mixed with the combustion gas stream prior to the introduction of the liquid hydrocarbon feedstock. Moreover, the amount of added water in the form of vapor found to be critical for the successful operation of the process ranges from 4 to 15 percent by volume based on the total gaseous volume of the fuel and oxidant utilized in preparing the primary combustion, with a preferred amount ranging from 4.6 to 11 percent and a particularly preferred range being from about 9 to about 11 percent by volume.

The following testing procedures are used in evaluating the analytical and physical properties of the blacks produced by the present invention.

Iodine Surface Area

The iodine surface area of a carbon black is determined in accordance with the following procedure and reported in units of square meters per gram (m^2/g). A carbon black

sample is placed into a size 0 porcelain crucible equipped with a loose-fitting cover to permit escape of gases and is devolatilized or calcined at a temperature of 1700°F (927°C) over a period of 7 minutes. The crucible and contents are then cooled in a dessicator, following which the top layer of calcined carbon black to a depth of about one-fourth inch is removed and discarded. From the carbon black remaining in the crucible a convenient sample is weighed, accurate to within 0.1 milligram (mg) and then transferred into a four ounce oil sample bottle. It has been found that for carbon blacks expected to have surface areas in the range of 300 to 750 m²/g an appropriate sample size is 0.1 gram whereas for blacks having surface areas in excess of 750 m²/g, an appropriate sample size is 0.05 gram. To the bottle containing the carbon black sample there is added 40 milliliters (ml) of 0.0473N iodine solution. The bottle is covered and the contents are then shaken for ten minutes at a rate of 120 to 260 back and forth trips per minute. The resulting solution is immediately centrifuged at a rate of 1200 to 2000 revolutions per minute (rpm) until the solution becomes clear, this usually covering a period of 1 to 3 minutes. Immediately after centrifuging, a 25 ml aliquot of the iodine solution, to which has been added a few drops of 1% starch solution as an end point indicator, is titrated with 0.0394N sodium thiosulfate solution until one drop of the sodium thiosulfate solution causes the blue color to become colorless. As a blank, 40 ml of the 0.0473N iodine solution is shaken, centrifuged and titrated in the same manner as above for the black-containing solution. The iodine surface area, expressed in m²/g, is calculated in accordance with the formula:

$$S.A. = \frac{10 (B-T) - 4.57}{1.3375}$$

Wherein B is the titration of the blank and T is the titration of the sample.

Dibutyl Phthalate (DBP) Absorption Number

The DBP absorption number of a carbon black, in pelleted form, is determined in accordance with ASTM Test Method D 2414-76.

Tint Strength

The tint strength of a carbon black sample is determined relative to an industry tint reference black in accordance with ASTM D 3265-76a.

pH Value of Carbon Black

Into a suitable Erlenmeyer flask there are placed a 5 gram sample of pelleted carbon black and 50 ml of distilled water. The carbon black-containing water mixture is brought to a boiling point using an electric hot plate, and maintained at a slow boil for a period of 10 minutes but not such as to cause dryness to occur. The resulting mixture is cooled to room temperature and the pH thereof is then determined utilizing a pH meter equipped with glass and calomel electrodes having an accuracy of ± 0.05 pH units. Prior to determining the pH of the carbon black, the pH meter is calibrated against two buffer solutions, one having a pH of 4.0 and the other having a pH of 7.0.

In order to evaluate the performance characteristics of the blacks in imparting compound moisture absorption and volume resistivity properties, the blacks are compounded with

a suitable resin, such as, ethylene/ethyl acrylate copolymer in the present instance. The compound to be tested is prepared by incorporating the desired amount of black into the resin, on a weight basis. For example, compounds containing carbon black in amounts of 12% by weight, 20% by weight and 36% by weight are commonly preferred for such evaluations.

The procedure for preparing the resin/black compounds involves adding one-half of the ethylene/ethyl acrylate resin to be used into a Banbury mixer, followed by the total amount of carbon black and then the remainder of the resin. The temperature of the Banbury mixer is brought to 100°F (37.8°C) and the mixing is commenced. The initial mixing is carried out for 30 seconds at 77 rpm (No. 1 speed) under a ram pressure of 40 p.s.i. (0.276 M Pa). Thereafter, the speed is increased to 115 rpm (No. 2 speed) for a period of 45 seconds. During this cycle the temperature reaches 100°F (37.8°C) whereupon the ram is raised to permit the black to be brushed back into the hopper. When the temperature reaches 250°F (121°C), water is circulated through the mixer housing and rotors. Following the period of mixing at 115 rpm, the speed is increased to 230 rpm (No. 3 speed) for an additional 105 seconds. At the end of this period of time, the mixing is stopped and the resin/black compound is removed from the mixer. In the case of a compound containing 12 or 20% by weight loading of the black, the temperature of the compound is from 260°F (127°C) to 290°F (143°C). Whereas for a 36% loading of black the compound temperature is from 330°F (166°C) to 360°F (182°C). The resulting compound is then passed twice through a cold two-roll mill and formed into sheets for the subsequent testing.

Compound Moisture Absorption

Sheets of the various ethylene/ethyl acrylate compounds prepared in the Banbury mixer as described above were subjected to dicing and granulating to yield suitable test samples. A two gram sample of the granulated compound is weighed into a glass crucible of known weight and dried overnight at 190°F (87.8°C) to remove any moisture in the compound. After cooling in a desiccator, the weight is obtained to the nearest one-tenth of a milligram. The compound is then placed into a desiccator maintained at conditions of room temperature and 79% relative humidity. The compound is then weighed after 1 hour and periodically thereafter for 3 days and further, if needed, until constant weight is achieved. The equilibrium moisture absorption is calculated as a weight percentage of the compound.

Volume Resistivity

This test procedure is employed for determining the volume resistivity of plastic compounds containing carbon black. The following is a description of the preparation from the sheeted compounds prepared in the Banbury mixer as described earlier of the compression molded plaques to be used as test specimens. From the sheeted compound prepared on the two-roll mill there are cut 7 x 7 inch (17.8 x 17.8 cm) samples. A compression mold with a cavity of 7 x 7 inch (17.8 x 17.8 cm) in size then lined with a release layer of polyethylene terephthalate film onto which is placed the test specimen. As a top release covering there is provided a film of polytetrafluoroethylene film. The covered compression mold is placed in a compression press which is maintained at a temperature of 320°F (160°C), as for example, by introducing

steam at a pressure of 100 p.s.i. (0.689 MPa). When the compression mold reaches a temperature of 320°F (160°C), the ram force of the compression press is raised from zero to a reading of 20 tons (18,144 kg) and is so maintained for a period of 5 minutes. The pressure on the sample is approximately 816 p.s.i. (5.63 MPa). The compression mold is then removed from the hot compression press and placed into a cold compression press, also maintained at 20 tons (18,144 kg) ram force, until the molds are cooled to about room temperature. The compression molded plaque of 7 x 7 inch (17.8 x 17.8 cm) size is then removed from the mold and deflashed.

To now prepare the actual specimen for the volume resistivity test procedure, a specimen of 2 x 6 inch (5.1 x 15.2 cm) size is cut from the 7 x 7 inch (17.8 x 17.8 cm) compression molded plaque. The test specimen is then coated on each end with a silver paint (silver conductive coating in ethyl alcohol) to produce an approximately one-half inch wide silver electrode. After drying, the uncoated portion of the specimen is measured to determine the exact distance between the electrodes, the average width and the average thickness. The specimen is then placed between 8 x 6 inch (20.3 x 15.2 cm) glass plates arranged crosswise to each other such that the edge of the top plate is evenly lined with the edge of the specimen. Brass shims are then placed on the top and bottom of each of the coated ends of the specimen. To the brass shims there are then attached alligator clips which lead to a Digitac Model H102120 Multimeter instrument for resistance measurements.

The resistance of the specimen is first measured into an oven maintained at 90°C in order to obtain a resistance measurement at this temperature. In so doing, the resistance

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is initially measured after 3 minutes at 90°C with subsequent readings being taken at 2 minute intervals for the next 30 minutes. After 30 minutes, readings are taken every 5 minutes until the specimen has been in the 90°C oven for a total of 60 minutes. The value for the resistance of the specimen at 90°C is fixed on a plot as the point where the readings become constant. The resistance measurements are then used in calculating the volume resistivity of the specimen by means of the following formula:

$$\text{Volume Resistivity, ohms-cm} = \frac{R \times A}{L}$$

Wherein R is the resistance of the specimen (ohms),

A is the cross-sectional area of the uncoated portion of the specimen (cm²), and

L is the distance between the two silver electrodes coated on each end of the specimen (cm).

The invention will be more readily understood by reference to the following examples which describe the detailed preparation of representative compounds. There are, of course, many other forms of this invention which will become obvious to one skilled in the art, once the invention has been fully disclosed, and it will accordingly be recognized that these examples are given for the purpose of illustration only, and are not to be construed as limiting the scope of this invention in any way.

Furthermore, as noted hereinbefore, there is a wide possibility of available materials from which to select a suitable liquid feedstock and fuel for combustion. However, in all of the examples herein, the same liquid feedstock and fuel were utilized. This in no way is intended to be limiting upon the materials that may be employed. As the liquid hydrocarbon feed-

stock there is used in the examples Sunray DX which is a fuel having by weight a carbon content of 90.4%, a hydrogen content of 7.56%, a sulfur content of 1.5%, an asphaltanes content of 4.4%, an ash content of 0.049%, a hydrogen to carbon ratio of 0.995, a sodium content of 2.8 ppm, a potassium content of 0.73 ppm, a B.M.C.I. of 135, a specific gravity in accordance with ASTM D 287 of 1.10, an API gravity of -3.1, an SSU viscosity (ASTM D 88) at 130°F (54°C) of 542.9 and on SSU viscosity at 210°F (99°C) of 63.3. The natural gas used as the fuel in all of the examples herein substantially comprises, by mole percent, 9.85% nitrogen, 0.18% carbon dioxide, 86.68% methane, 3.07% ethane, 0.19% propane, 0.01% isobutane, and 0.02% n-butane.

Example 1

In this example there is employed a suitable reaction apparatus provided with means for supplying combustion gas-producing reactants, i.e., a fuel and an oxidant, either as separate streams or as precombusted gaseous reaction products to the primary combustion zone, and also means for supplying both the carbon black-yielding hydrocarbonaceous feedstock, the combustion gases to be introduced downstream to the apparatus, means for introducing the additional amounts of water, etcetera. The apparatus may be constructed of any suitable material such as metal and either provided with refractory insulation or surrounded by cooling means such as a recirculating liquid which is preferably water. Additionally, the reaction apparatus is equipped with temperature and pressure recording means, means for quenching the carbon black-forming reactions such as spray nozzles, means for cooling the carbon black product and means for separating and recovering the carbon black from other undesired by-products.

In a more detailed description of the apparatus utilised herein, the first stage is employed so as to obtain a substantially completely pre-formed combustion prior to feedstock injection. As a suitable burner there is provided an enclosed reaction vessel having a diameter of 8.4 inches (0.213m) for a length of 26.6 inches (0.676m) which is then reduced conically over the next 5 inches (0.135m) to a diameter of 5.3 inches (0.135m). Connected to the first zone, or burner section, is a second zone referred to as the transition zone having a diameter of 5.3 inches (0.135m) and a length of 9 inches. It is in this zone that the liquid feedstock is injected as coherent streams through as many orifices as desired. The feedstock is injected under conditions sufficient to assure a proper degree of penetration into the combustion gas stream thereby avoiding problems of coke formation in the reactor. The resultant hot gas stream then enters a third zone, referred to as the reaction zone where the carbon black is formed. This zone extends to the point where the reaction is quenched. In the present case the reaction zone consists of a section having a diameter of 36 inches (0.914m) and a length of 16 feet (4.88m) followed by a section having a diameter of 27 inches (0.69m) and a length of 11 feet (3.35m).

Accordingly, in carrying out the present example combustion air, preheated to 710°F (377°C) is fed into the

combustion zone at a rate of 70,700 sc.f.h. ($0.526 \text{ m}^3/\text{s}$)
and the natural gas at a rate of 6540 s.c.f.h. ($0.0487 \text{ m}^3/\text{s}$)
under a pressure of 26 p.s.i.g. (0.179 MPa). Added water
is brought into the reactor with the combustion air in an
amount of 35 gph (6140 s.c.f.h.) ($0.0457 \text{ m}^3/\text{s}$) or 7.3% by
volume. Furthermore, oxygen is added at a rate of 7000
s.c.f.h. ($0.0520 \text{ m}^3/\text{s}$) to the combustion chamber. Such
conditions result in a primary combustion of 180% and a
combustion chamber pressure 5.1 inches (17.2kPa) of mercury.
The resultant stream of hot combustion gases flow into the
transition zone where the coherent jets of liquid feed-
stock are injected thereinto from the periphery through
four openings, each of 0.038 inch (0.97mm) in size. The
feedstock is preheated to 400°F (204°C) and is fed at a
rate of 101 gph (0.117 kg/s) under a pressure of 216 p.s.i.g.
(1.49 MPa). The residence time of

the reaction is 0.7 second and the gas stream is quenched with water to a temperature of 1400°F (760°C). The overall percent combustion of the process is 48.1%. The black is collected normally and then pelletized and dried under oxidizing conditions of a sufficient nature as to yield a black having an iodine surface area of 740 m₂/g, a pH of 3.2, a DBP of 197 cc/100g, and a tint strength of 132%. Further information appears in Table III.

Example 1

The procedure and apparatus used herein are similar to that of Example 1 with certain exceptions. Combustion air, preheated to 710°F (377°C), is introduced into the combustion chamber at a rate of 71,100 s.c.f.h. (0.529 m³/s) containing mixed therein with water introduced at a rate of 35 gph (6140 s.c.f.h.) (0.0457 m³/s) which is 7.9% by volume based on the total volume of reactants utilized to prepare the primary combustion. The natural gas is fed into the first zone at a rate of 3340 s.c.f.h. (0.0248 m³/s) under a pressure of 10 p.s.i.g. (0.069 MPa). In this instance oxygen is also added into the combustion zone at a rate of 3500 s.c.f.h. (0.0260 m³/s). Under these conditions, there is obtained a primary combustion of 298.7% and the pressure of the combustion chamber is found to be 3.2 inches (10.8 kPa) of mercury. The liquid feedstock, preheated to 405°F (207°C), is then injected into the hot, high velocity stream of combustion gases, in the form of coherent jets or streams, through four (4) unobstructed openings each of which is 0.036 inch (0.91 mm) in size. The liquid feedstock is injected at a rate of 99 gph (0.115 kg/s) and under a pressure of 245 p.s.i.g. (1.69 MPa). The third stage, or reaction zone, is composed of a section having a length of 24 feet (7.31 m) and a diameter of 36 inches (0.91 m)

followed by a section having a length of 9 feet (2.74 m) and a diameter of 27 inches (0.686 m). Following a reactor residence time of 1 second, the reaction is water-quenched to a temperature of 1400°F (760°C). The overall combustion of the process is 47.7%.
5 The analytical and physical properties of this black are given in Table III.

Example 3

Utilizing the procedure and apparatus of Example 1 with minor modifications, the following run is carried out.
10 Combustion air, preheated to 705°F (374°C), is introduced into the first zone of the reaction apparatus at a rate of 71,000 s.c.f.h. (0.528 m³/s) containing entrained therein added water which is introduced at a rate of 35 gph (6140 s.c.f.h.) (0.0457 m³/s) or 7.7% by volume based on the gaseous volume of
15 reactants employed in preparing the primary combustion. There are also fed into the combustion chamber natural gas at a rate of 5540 s.c.f.h. (0.0412 m³/s) under a pressure of 21 p.s.i.g. (0.145 MPa) and oxygen at a rate of 3500 s.c.f.h. (0.0260 m³/s). This results in a primary combustion of 179.6% and the pressure
20 of the combustion chamber is measured as 4.1 inches (13.3 kPa) of mercury. The resultant gaseous stream enters the transition zone (9 inches (0.229 m) in length by 5.3 inches (0.134 m) in diameter) and the liquid feedstock, preheated to 405°F (207°C), is injected therein, as solid streams, through four (4) un-
25 obstructed orifices of 0.040 inch (1.02 mm) size, at a rate of 86 gph (0.100 kg/s) under a pressure of 140 p.s.i.g. (0.97 MPa) thereby obtaining proper penetration to assure good atomization and dispersion of the feedstock. The gaseous stream enters the reaction chamber which is formed of 2 sections, one having a
30 length of 24 feet (7.32 m) by a diameter of 36 inches (0.91 m)

and the subsequent having a length of 3 feet (2.44 m) and a diameter of 27 inches (0.686 m), therein, after a residence time of 1 second, the reaction is water-quenched to 1400°F (760°C). Thereafter, the black is collected and pelletized and dried under oxidizing conditions. The overall percent combustion of the process is 47.9%. Analytical and physical properties of this black are reported in Table III.

Example 8

Essentially similar apparatus and procedure as used in Example ³ 8 is employed herein except as follows. Combustion air, preheated to 750°F (399°C), is introduced into the combustion chamber at a rate of 70,700 s.c.f.h. (0.526 m³/s) containing added water injected therinto at a rate of 20 gph (3510 s.c.f.h.) (0.0261 m³/s) which is determined to be 4.5% by volume of first stage reactants. The natural gas is injected into the first stage at a rate of 6700 s.c.f.h. (.05 m³/s) under a pressure of 26 p.s.i.g. (0.179 MPa). Under such conditions, the primary combustion is 119.3% and the pressure in the combustion chamber is 3.6 inches (12.2 kPa) of mercury. The hot stream of gaseous combustion products then enters the transition zone where liquid feedstock, preheated to 330°F (166°C), is injected therinto from the periphery under a pressure of 320 p.s.i.g. (2.20 MPa). The feedstock is injected through four (4) unobstructed openings each of which is 0.0225 inch (0.57 mm) in size at a rate of 44 gph (0.051 kg/s). Following a residence period of 1.0 second, the reactor is water-quenched to a temperature of 1400°F (760°C). The overall percent combustion of the process is 55.5%. The black is collected and subjected to pelletizing and drying under oxidizing conditions so as to yield a low pH product. The analytical and physical properties are reported in Table III.

Example 10⁵

The procedure and apparatus of Example ⁴ are followed except as noted. Combustion air, preheated to 710°F (377°C), is fed into the combustion chamber at a rate of 60,500 s.c.f.h. (0.450 m³/s) together with added water entrained therein at a rate of 31 gph (5440 s.c.f.h.) (0.0405 m³/s), or 8.3% by volume based on the total volume of the combustion air and natural gas used to produce the primary combustion. Natural gas is introduced into the first zone at a rate of 4910 s.c.f.h. (0.0365 m³/s) under a pressure of 17 p.s.i.g. (0.117 MPa). The primary combustion is determined to be 139.7% and the combustion chamber pressure is read as 2.7 inches (9.1 kPa) of mercury. The hot stream of gaseous products then passes into the transition zone equipped with four (4) unobstructed orifices around the periphery each of 0.025 inch (0.64 mm) in size. The liquid feedstock, preheated to 330°F (166°C) is injected into the gaseous stream through the four orifices at a rate of 41 gph (0.043 kg/s) under a pressure of 167 p.s.i.g. (1.15 MPa). The residence time of the reaction is 1.2 seconds prior to quenching with water to a temperature of 1400°F. The overall percent combustion is 55.9%. The black is collected, and pelletized and dried under oxidizing conditions such that a black of low pH is produced. The analytical and physical properties of the black are given in Table III.

Example 11⁶

Following closely the procedure of Example ⁵ 10, combustion air, preheated to 735°F (391°C), is introduced into the combustion chamber at a rate of 70,100 s.c.f.h. (0.522 m³/s) containing therein added water introduced at a rate of 46 gph (8070 s.c.f.h.) (0.060 m³/s) which corresponds to 10.5% by volume based on the total volume of air and gas used to produce the

TABLE I

Black Sample	Ex. 1	Ex. 2	Ex. 3	Ex. 4	Ex. 5	Ex. 6
Iodine Surface Area m ² /g	740	630	772	865	934	1029
pH	3.2	3.6	3.4	3.0	3.3	4.0
PBP, cc/100g	197	196	225	241	264	274
Tant Strength %	132	121	138	134	142	148
Polyene/Ethyl acrylate Resin Containing 12% Loading Carbon Black (By weight)						
Volume Resistivity, Room Temp., ohm-cm	-	101	49	30	22	18
Volume Resistivity, 90°C, ohm-cm	-	131	57	29	22	17
Compound Moisture Absorption %	-	0.56	0.58	0.89	0.44	0.38

primary combustion. The natural gas is introduced into the first zone at a rate of 6750 s.c.f.h. ($0.950 \text{ m}^3/\text{s}$) under a pressure of 18 p.s.i.g. (0.124 MPa). As a result, there is produced a primary combustion of 119.9% and a combustion chamber pressure of 3.1 inches (10.4 kPa) of mercury. The resultant hot stream of water-laden combustion gases enters the transition zone where liquid feedstock, preheated to 340°F (154°C), is injected thereinto through four (4) unobstructed orifices of 0.0225 inch (0.57 mm) size peripherally. The feedstock is fed at a rate of 41 gph (0.048 kg/s) and under a pressure of 170 p.s.i.g. (1.17 MPa) thereby assuring proper penetration of the combustion gas stream. The residence time in the reactor is 1.0 second followed by water-quenching of the reaction to a temperature of 1345°F (729°C). The overall percent combustion of the process is 57%. The black is collected, pelletized and dried under oxidizing conditions. Further details of the black of this example appear in Table III.

From the above data it is apparent that the process of the present invention results in the preparation of furnace blacks having exceptionally high surface areas. Moreover, the data indicate that the conductivity properties of the furnace process blacks of this invention are reasonably comparable to that of the highly conductive gasification process by-product blacks.

While this invention has been described with respect to certain embodiments, it is not so limited, and it should be understood that variations and modifications thereof may be made which are obvious to those skilled in the art without departing from the spirit or scope of the invention.



claim 5

1. A furnace-type carbon black product characterised by having an iodine surface area of at least $600\text{m}^2/\text{g}$, a pH value of less than 5 and a DBP value of at least 160cc/100 g.
2. A furnace-type carbon black product according to claim 1 wherein the iodine surface area is at least $800\text{m}^2/\text{g}$.
3. A furnace-type carbon black product as claimed in claim 1 or claim 2 wherein the iodine surface area is in the range of 800 to $1100\text{m}^2/\text{g}$.
4. A furnace-type carbon black product as claimed in any one of claims 1, 2 or 3 wherein the pH is in the range 2 to 4.
5. A furnace-type carbon black product as claimed in claim 4 wherein the pH is in the range 3 to 4.
6. A furnace-type carbon black product as claimed in any one of claims 1 to 5 wherein the DBP value is in the range 180 to 350 cc/100g.
7. A furnace-type carbon black product as claimed in claim 2 wherein the DBP value is in the range 180 to 275 cc/100g.
8. A furnace-type carbon black product as claimed in claim 5 wherein the DBP value is in the range 180 to 275 cc/100g.
9. A furnace-type carbon black product as claimed in claim 1 and substantially as described in examples

1-6.