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(54) Title: METHOD OF REFINING CARBON BLACK CHAR

(57) Abstract: A method of refining carbon black char derived from the pyrolysis of scrap tyres. The method comprises a pyrolysis step wherein the carbon black char is pyrolysed to produce a pyrolysed carbon black char feed.
METHOD OF REFINING CARBON BLACK CHAR

The present invention relates to methods of refining carbon black char resulting from the pyrolysis of carbonaceous products, and to uses of refined and purified carbon black char, such as a pigment, an ink jet carbon black dispersion or formulation, and an ink jet ink. The present invention extends to apparatus for refining carbon black char.

Passenger cars, trucks, lorries etc. wear out many millions of tyres every year. A high proportion (approximately 25% to 35%) of the content of tyres is carbonaceous being derived from carbon black, which is a finely divided form of carbon. The carbon black is added to tyres together with steel wire to reinforce and strengthen the tread and side walls thereof. Scrap tyre material may be decomposed at temperatures typically in the range of between 600°C and 800°C. This decomposition process is known as pyrolysis, and results in the production of gas, oil, and impure carbon black char which consists of steel wire, fused silica particles, free sulphur, and also other non-carbon elements.

It has been found through analysis, that the carbon black char produced by pyrolysis of carbonaceous materials contains many impurities including metallic salts, which render the material unsuitable for some applications including desk jet and ink jet dispersions, and finished ink jet printer inks. Small quantities of other impurities including various compounds, silica and sulphur, are not desirable in most ink applications because they displace the carbon, which results in a low carbon concentration,
which affects the final properties of the ink. Furthermore, these impurities are also more difficult to mill down to acceptable particle sizes than pure carbon which results in extended processing times and, therefore, increased cost in the manufacture of the ink. Another problem involved with using carbon black char made from pyrolysis of scrap tyres is that, the fragments of steel wire, which are produced, have to be removed from the pyrolysis by-product before the carbon black char can be processed in to ink jet printer ink, and ink jet carbon black dispersion.

The impure carbon black char that results from the pyrolysis of scrap tyre material also comprises hydrocarbon compounds including aromatic and short chain hydrocarbons. Such compounds have been found to have an undesirable effect on the refined and purified carbon black. For example, a carbon black formulation or dispersion made from the carbon char may be excessively viscous. The ink jet ink produced from the formulation can block a printer cartridge.

In contrast to impure carbon black char recovered from scrap tyre pyrolysis, hydrocarbon-derived virgin carbon black, which is the overwhelming world source of finished black inks and dispersions, has far fewer impurities. However, problems associated with using virgin carbon black in ink dispersions include re-agglomeration of the carbon particles. Re-agglomeration results in poor dispersion of the ink when printing.

In addition, environmental considerations are beginning to force the ink jet industry to search for more
environmentally friendly processes and substitute substances, which could be used instead of virgin carbon black. The inkjet industry is moving from solvent to aqueous based systems, and from soluble dyes to pigments. A key disadvantage of using pigments is in stabilising the material so that it does not agglomerate and increase in particle size, which then settle out of solution. This settling of the pigment causes terminal failures in the small channels and nozzle orifices of an inkjet print head.

It is an aim of embodiments of the present invention to provide a method of refining impure carbon black char. Particularly, it is an aim to provide a method of refining carbon black char derived from the pyrolysis of scrap tyres. In addition, it is an aim to provide a method for producing a pigment derived from impure carbon black char. The impurities from the impure carbon black char may be removed because then the product would be directly saleable into the ink jet industry as an input material for ink jet carbon black dispersions and finished ink jet printer inks. Milling is undertaken to reduce particle size in high concentration ink jet dispersions (typically 15% solids or greater), and this concentration is "let-down" or diluted to approximately 5% solids within the finished ink formulation. Therefore, it would also be advantageous to produce a unique formulation that can be developed to counter agglomeration and foaming in milling and "let-down", and to improve stability in the storage thereof prior to use and in let-down, and finished inks.

According to a first aspect of the present invention, there is provided a method of refining carbon black char,
the method comprising removing particles greater than approximately 1mm in size from a carbon black char feed.

By the term "refining", we mean that carbon present in the feed is extracted or purified from the feed. Preferably, the particles greater than approximately 1mm in any dimension, for example, length, width, diameter etc., are separated from the char feed. Preferably, particles which are greater than 1mm in size are separated as waste, and disposed of. Preferably, the removal of particles greater than 1mm in size produces a pigment comprising carbon, which is preferably a high purity carbon pigment. Preferably, the pigment comprises carbon black. Preferably, the pigment comprises at least 50% (w/w) carbon, more preferably, at least 65% (w/w) carbon, even more preferably, at least 80% (w/w) carbon, and most preferably, at least 95% (w/w) carbon. In a preferred embodiment, the pigment comprises at least 98% (w/w) carbon.

In a further preferred embodiment the pigment comprises at least 99% (w/w) carbon.

 Preferably, the carbon black char is derived from the pyrolysation of carbonaceous material, most preferably, scrap tyres. Preferably, the carbon black char is contained within pyrolysis by-products. Hence, advantageously, the method is an environmentally friendly way of producing a high purity carbon black pigment from scrap tyres.

 Preferably, the method comprises classifying the carbon char feed by particle size. Approximately 10% of a tyre,
and about 30% of carbon char derived from pyrolysis of tyres, is steel wire. In addition, a large proportion of the carbon char feed consists of part- or un-pyrolysed materials, for example, steel, rubber, and also compounds of silica, which form glass beads, and fibreglass. The majority of these materials tends to be larger than 1mm across. Preferably, and advantageously, the method removes these particles greater than 1mm in size.

Preferably, the method comprises classifying the carbon char feed by size such that at least 50%(w/w) of the feed has a particle size of less than approximately 1mm. Preferably, the size classification step removes particles larger than 1mm so that at least 60%(w/w), more preferably 70%(w/w), even more preferably 80%(w/w), and most preferably 90%(w/w), of the feed has a particle size of less than approximately 1mm.

The particles greater than 1mm may be removed from the carbon char feed by screening. Preferably, the screening comprises vibration. The screening may comprise use of a locker screen, or the like.

Preferably, the method further comprises the step of magnetically separating impurities from the carbon black char feed. Preferably, the magnetic separation step is carried out directly after the pyrolysis of the carbonaceous materials, preferably, scrap tyres, preferably before the size classification step. Preferably, the method comprises varying a magnetic field in the vicinity of the carbon char feed to which the magnetic impurities are attracted, thereby separating the magnetic impurities therefrom. Preferably, the magnetic
separation step comprises use of a magnetic screen, which screen preferably comprises at least one bar magnet and, preferably, at least one rare earth magnet, to which the metallic materials are attracted.

Preferably, and advantageously, this step removes metallic and other magnetically attractive impurities, for example, fragments of steel wire present in the carbon char feed. Preferably, and advantageously, the carbon black char feed is substantially dry, preferably, comprising a dry powder.

Disadvantageously, known methods for removing magnetic impurities from impure carbon black char comprise reacting them with acid/alkali, which may be weak. Such methods involve the use of hazardous chemicals and take a substantial period of time to allow the chemical reaction to complete. Advantageously, use of a magnetic field in the present invention is a quicker and safer way of removing the magnetic impurities from the carbon char feed, allowing the feed to remain dry which is easier to work with than acid/alkali solutions.

Alternatively, the carbon char feed may be reacted with acid/alkali. The carbon black produced from reacting with acid/alkali is highly pure, preferably at least 98% w/w pure carbon. This level of purity is a requirement of some end users of the carbon black product, particularly manufacturers of inkjet ink.

Preferably, the method comprises a second size classification step, which may comprise milling and/or grinding the feed. Suitably, the method comprises removing particles from the feed, which are greater than
approximately 800μm in size, more suitably, greater than 600μm in size, even more suitably, greater than 400μm in size. Preferably, the method comprises removing particles from the feed which are greater than 200μm in size, more preferably, greater than 100μm in size, even more preferably, greater than 50μm in size, and more preferably, greater than 20μm in size.

In an alternative and preferred embodiment, the method comprises a second size classification step for removing particles from the feed which are greater than 5μm. Advantageously, particles less than 5μm provide a large surface area for treatment for impurity removal. The process prepares the carbon for surface treatments.

Preferably, the second size classification step removes particles from the feed so that at least 80%(w/w), more preferably 90%(w/w), even more preferably 95%(w/w), and most preferably 98%(w/w), of the feed has a particle size of less than approximately 800μm, preferably, less than 600μm, preferably, less than 400μm, more preferably, less than 200μm, more preferably, less than 100μm, more preferably, less than 50μm and, preferably, less than 20μm, most preferably less than 5μm. Preferably, the second size classification step takes place in a substantially closed environment, in order to capture fugitive airborne particulate emissions.

Preferably, the second size classification step removes particles from the carbon char feed so that at least 99%(w/w) of the feed has a particle size of less than 5μm.
Preferably, the method comprises classifying the carbon char feed by density. Suitably, the method comprises removing particles from the feed which have a density greater than approximately 2.0g/cm³, more suitably, greater than 1.5g/cm³, and more suitably, greater than 1.2g/cm³. Preferably, the method comprises removing particles from the feed which have a density greater than approximately 1.0g/cm³, more suitably, greater than 0.75g/cm³, and more suitably, greater than 0.5g/cm³.

The density of purified carbon in the pigment is approximately 0.44g/cm³, which was calculated by filling a 100cm³ box with purified carbon pigment which was not compacted, and then weighed.

Preferably, the size classification and the density classification steps are carried out by the same apparatus, such as an Opposed Jet Classifier Mill. Preferably, and advantageously, the size and density classification steps remove part- or un-pyrolysed materials, and compounds of silica. Known methods for refining impure carbon black char do not have a step for removing unpyrolysed materials, such as rubber, fibre glass etc. Preferably, overdense particles are removed from the feed retained in the mill.

Preferably, the method comprises removing sulphur compounds and preferably, metallic salts, from the impure carbon black char feed. Preferably, the carbon black char feed is mixed with water at a ratio in the range of 1:0.5 to 1:1.5. Preferably, the water is deionised, and preferably produces a carbon black slurry.
Preferably, the carbon black/water slurry is maintained at a temperature of at least 50°C, more preferably, 60°C, and most preferably, 70°C.

Preferably, the carbon black/water slurry is maintained at this temperature for at least 15 min, more preferably, at least 30 min, even more preferably, at least 45 min, and most preferably, at least 1 hr.

Preferably, the method comprises a step for removing water from the slurry such that the concentration of water in the resultant slurry is less than approximately 50% (w/w), more preferably, less than 25% (w/w), even more preferably, less than 15% (w/w) and most preferably, less than 10% (w/w). Preferably, the de-watering step is carried out in a centrifuge.

Preferably, the method comprises mixing water with the resultant carbon black slurry, preferably at a ratio in the range of 1:0.5 to 1:1.5. Preferably, the water is deionised or demineralised. Preferably, the method comprises dewatering the resultant slurry to produce a high purity carbon black pigment, which comprises a water concentration of less than approximately 50% (w/w), more preferably, less than 25% (w/w), even more preferably, less than 15% (w/w) and most preferably, less than 10% (w/w).

In a most preferred embodiment, the method of refining carbon-black char derived from the pyrolysis of scrap tyres, comprises a step of pyrolysing the carbon black char to produce pyrolysed carbon black char feed. Preferably, the pyrolysis of the carbon black char feed
takes place after the carbon black char feed has passed through a size classification step or a density classification step. Preferably, the pyrolysis of the carbon black char feed takes place after a screening step or magnetic separation step. Most preferably, the pyrolysis of the carbon black char feed takes place after the steps of

a) screening the carbon black char feed derived from pyrolysis of scrap tyres;

b) magnetic separation

c) size classification, which may comprise milling and/or grinding the carbon black char feed;

d) density classification of the carbon black char feed.

The pyrolysis of the carbon black char feed reduces the content of aromatic compounds and hydrocarbon compounds in the resultant pyrolysed carbon black char feed and converts unpyrolysed and part pyrolysed material to carbon and ash.

In particular, the pyrolysis of the carbon black char feed reduces the sulphur content of the pyrolysed carbon black char feed. The effect of this is to produce a low viscosity ink dispersion. The ink formulation made from the low viscous dispersion enables a high quality print to be produced which is most desirable to the user.

Furthermore, the pyrolysis of the carbon black char feed improves the wettability of the resultant pyrolysed carbon black char which significantly reduces the quantity of wetting dispersant needed. Advantageously, the cost of carrying out the process is reduced. The process produces
a less viscous dispersion which formulates in not an ink which has excellent print quality characteristics. Additionally, an improvement in colour density of the ink is evident due to the reduced amount of dispersion.

Preferably, the pyrolysis of the carbon black char takes place in a chamber having a one-way valve. In this manner, gas generated by said pyrolysis is allowed to exit the chamber whilst air is prevented from entering the chamber.

Preferably, the pyrolysis of the carbon black char is maintained at a temperature of at least 650°C, more preferably, 800°C. In a most preferred embodiment, the temperature is maintained at 800°C for at least 5 hours.

In a preferred embodiment, the method comprises a first washing step wherein the pyrolysed carbon black char is washed with a first washing liquor. Preferably, the first washing liquor is acidic solution, preferably, a hydrochloric acid solution. Preferably, the method further comprises a second washing step wherein the pyrolysed carbon black char is washed with a second washing liquor. Most preferably, the second washing liquor is an alkaline solution.

Advantageously, washing the pyrolysed carbon char feed with an acidic solution significantly removes the % w/w of metals and sulphur still present in the further pyrolysed char.

Advantageously, washing the pyrolysed carbon black char with an alkaline solution significantly reduces the % w/w
of silicon in the feed. Silicon is a thickening agent which causes the ink to agglomerate and block the cartridge. Advantageous, the alkaline solution reduces this problem.

Additionally and advantageously, for a high purity carbon demanded by some users, washing the pyrolysed carbon black char with an acidic solution and an alkaline solution results in a carbon black char which is of suitable quality. The acid and alkali treatment stages may and are preferably, undertaken in tandem.

The steps making up the method may be carried out in any order.

In a most preferred embodiment the method comprises the steps of:-

a) Pyrolysis of scrap tyres to produce an impure carbon black char feed;
b) classification of the carbon black char feed;
c) pyrolysis of the carbon black char feed;
d) acid washing of the carbon black char feed;
e) alkali washing of the carbon black char feed;
f) demineralised water washing of the carbon black char feed; and
g) processing of the carbon black char feed to produce a carbon black product.

Preferably, step c) takes place before step d). Most preferably, step c) occurs after step b) and before step d).
Preferably, step g) comprises the steps of milling and dispersion of the carbon black char feed to produce a high quality ink jet ink. Advantageously, the ink jet ink produced is highly pure being at least 98% w/w carbon, preferably 99% w/w carbon.

According to a second aspect, there is provided a method of producing a pigment comprising carbon black, the method comprising removing particles greater than approximately 1mm in size from a carbon black char feed, to produce a carbon black pigment.

In a preferred embodiment, the method of producing a pigment comprising a carbon black further comprises pyrolysing the carbon black char feed to produce a pyrolysed carbon black char feed. More preferably, the method comprises washing the pyrolysed carbon black char feed with a first washing liquor. Preferably, the first washing liquor is acidic solution, preferably hydrochloric acid solution. Preferably, the pyrolysed carbon black char feed is washed with a second washing liquor, preferably an alkaline solution.

In a most preferred embodiment, there is provided a method of producing a pigment comprising carbon black, the method comprising removing particles greater than approximately 5μm in size from a carbon black char feed, pyrolysing the carbon black char feed to produce a pyrolysed carbon black char feed, washing the pyrolysed carbon black char feed with a first washing liquor and washing the pyrolysed carbon black char feed with a second washing liquor.
According to a third aspect, there is provided a pigment comprising carbon black derived from a feed of carbon black char, wherein particles greater than approximately 1mm in size are removed from the carbon black char feed.

In a preferred embodiment, there is provided a pigment comprising carbon black, wherein the carbon black is derived from pyrolysed carbon black char. Preferably, the pyrolysed carbon black char is washed with a first washing liquor and preferably, a second washing liquor.

Preferably, the feed comprises impure carbon black char. Advantageously, and preferably, the pigment is derived from recovered waste material or carbonaceous char from pyrolysis, for example, of scrap tyres as opposed to virgin carbon black, which has been cracked from hydrocarbons. In addition, advantageously, the pigment is produced without the use of harmful substances such as acids, alkali and solvents. Therefore, the pigment is produced by a method, which is environmentally friendly both in terms of raw material input and process. In addition, the removal of impurities results in a high purity carbon black pigment, which is comparable to virgin carbon black.

Alternatively, the pigment is produced with acid and alkali treatment for some end uses. Acid and alkali solution wastes from the processes are combined to produce PH neutral effluent for safe disposal. Thereby the pigment is produced by a method that retains environmentally friendly features.
According to a fourth aspect of the present invention, there is provided apparatus for refining carbon black char, the apparatus comprising means to remove particles greater than approximately 1mm in size from a carbon black char feed.

Preferably, the apparatus comprises feed means for feeding the carbon black char feed to the means for removing particles greater than 1mm.

Preferably, the apparatus further comprises means to apply a magnetic field to the carbon black char feed, to thereby magnetically separate magnetic impurities therefrom.

Preferably, the apparatus comprises size and preferably, density classification means, which are adapted to classify the feed in terms of size and density, respectively. Preferably, the apparatus comprises washing means adapted to substantially remove sulphur and, preferably metallic salts, from the feed.

In a preferred embodiment the apparatus comprises vessels for withstanding heat and containing carbon black char. Preferably, the vessels exclude the ingress of air whilst enabling the escape of gas generated by heat. Preferably the apparatus comprises heating and control means to maintain and control heat and measure and record temperature.

According to a fifth aspect, there is provided a composition comprising anti-foaming agent, surfactant, and carbon black, wherein the carbon black is derived from a feed of carbon black char, wherein particles greater than
approximately 1mm in size are removed from the carbon black char feed.

In a most preferred embodiment, there is provided a composition comprising anti-foaming agent, surfactant, and carbon black, wherein the carbon black is derived from pyrolysed carbon black char produced from the pyrolysis of scrap tyres.

Preferably, the surfactant comprises a dispersant.

Optionally, the composition comprises water, preferably at a concentration of approximately 30-60% (w/w) of the total composition, more preferably 35-55% (w/w), and most preferably 40-50% (w/w) of the total composition.

Preferably, the composition comprises approximately 10-70% (w/w) carbon black, more preferably, 20-50% (w/w) and, most preferably, 25-40% (w/w) carbon black of the total composition.

Preferably, the composition comprises approximately 0.1-10% (w/w) anti-foaming agent, more preferably, 0.15-5% (w/w), and most preferably, 0.2-3% (w/w) anti-foaming agent of the total composition.

Preferably, the composition comprises approximately 1-30% (w/w) surfactant, more preferably, 5-20% (w/w), and most preferably, 10-15% (w/w) surfactant of the total composition.

Suitably, the composition is treated to remove particles, which are greater than 75μm in size, more suitably,
greater than 50μm in size, and even more suitably, greater than 25μm in size, from the composition. Preferably, the composition is treated to remove particles, which are greater than 15μm in size, even more preferably, greater than 5μm in size, and most preferably, greater than 2μm in size, from the composition. In a preferred embodiment, particles greater than 0.5μm are removed from the composition.

Preferably, the particle size of the composition is in the range of approximately in the range of 0.001μm-1.000μm, more preferably, approximately 0.010μm-0.750μm, and most preferably, 0.060μm-0.500μm.

Preferably, the said treating comprises micronising, preferably by a microniser, preferably to produce a dispersion. The micronising means may be a Buhler ZR120+.

The carbon black, pigment, composition, and/or dispersion defined in the above aspects may be used for the manufacture of ink, for example, ink jet and desk jet printer ink, inks including flexographic, offset, ultra violet cured, heatset, and coldest. Further applications include the use in the manufacture of toner, including laser printer toner and photocopyer toner. Other applications include the use in paints, powder coating and timber treatment.

According to a sixth aspect, there is provided an ink formulation comprising the composition defined in the fifth aspect, ethylene glycol, and diethylene glycol.
The ink formulation may be used for the manufacture of an ink jet printer ink.

Optionally, the formulation comprises water, preferably at a concentration of approximately 1-50% (w/w) of the total formulation, more preferably 10-40% (w/w), and most preferably 25-55% (w/w) water of the total formulation.

Preferably, the formulation comprises approximately 10-80% (w/w) composition defined in the fifth aspect, more preferably, 25-60% (w/w) and, most preferably, 40-50% (w/w) of the composition defined in the fifth aspect of the total formulation.

Preferably, the formulation comprises approximately 1-30% (w/w) ethylene glycol, more preferably, 5-15% (w/w) and, most preferably, 8-12% (w/w) ethylene glycol of the total formulation.

Preferably, the formulation comprises approximately 1-30% (w/w) diethylene glycol, more preferably, 5-15% (w/w) and, most preferably, 8-12% (w/w) diethylene glycol of the total formulation.

All of the features described herein may be combined with any of the above aspects, in any combination.

For a better understanding of the invention, and to show how embodiments of the same may be carried into effect, reference will now be made, by way of example, to the accompanying diagrammatic drawings, in which:-
Figure 1 is a schematic flow chart showing a process for refining impure carbon black.

Figure 2 is a schematic flow chart showing a process for refining impure carbon black according to a second embodiment of the invention.

Figure 3 is a simple schematic flow chart showing a step included in the second embodiment.

Figure 4 is a simple schematic flow showing a further step included in the second embodiment.

Figure 5 is a simple schematic overview of a method according to the second embodiment.

Referring to Figure 1, there is shown a flow chart of a process 2 for refining impure carbonaceous char into a dispersion, which can be used to produce ink jet printer ink, and toner. Waste tyres are initially fed 4 into a pyrolysis unit 6 to produce impure carbon black char 7. The char 7 is then screened using a magnetic screen 8 and a locker screen 9 (section 1). The magnetic screen 8 removes ferrous material impurities 18 from the carbon char, and the locker screen 9 removes particles in the char which are greater than 1mm in size.

The char particles are then further classified using an Opposed Jet Classifier Mill 12, which extracts smaller particles of metal, and grinds the char down such that the average particle size is no greater than 20μm (section 2). The char particles are sucked under vacuum out of the Classifier mill 12 by a cyclone 22 (section 3), after
which metallic salts and sulphur are extracted, followed by two cycles of dewatering to produce a carbon char slurry (section 4).

Following the dewatering stages, the char slurry is sent to a holding tank 42 where it is mixed with demineralised water to produce a high purity carbon black slurry (section 5). The high purity carbon black slurry is then processed to produce a dispersion, which can be used in the manufacture of paints, ink jet printer inks and toner (section 6). The dispersion is then filtered and sent to a post-processing holding tank 74 (section 7), to await quality control (section 8), and then final processing to produce an ink formulation (section 9). Finally, the products are packaged and then dispatched (section 10). Each stage of the refining process will now be described in detail from Section 1 through to Section 9 shown in Figure 1.

Referring to Section 1 of Figure 1, a source of scrap vehicle tyres 4 is initially fed into a pyrolyssation machine 6, which heats the tyres to temperatures in the range of 600°C-800°C. The tyres decompose producing a source of impure carbon char 7, which consists of steel wire, fused silica particles, free sulphur, and also other non-carbon elements. The carbon char material 7 is variable in size and includes particles as large as 4mm in size.

The char material 7 is taken from the pyrolysis unit 6 and is fed into a magnetic screen 8 (British Rema, Dronfield, UK), which removes larger metallic particles such as steel wire. The magnetic screen 8 consists of a number of bar
and rare earth magnets (not shown) are arranged around a conveyor system. The magnets attract and thereby extract magnetic metals and other ferrous materials out of the carbon char material 7. The magnets are cleaned periodically of the ferrous and other magnetic material, which is collected and safely disposed.

Following removal of the metallic particles, the carbon char is then fed to a vibrating locker screen 9 (British Rema, UK), which removes particles 18 greater than 1mm in diameter, which are sent to waste.

Referring to Section 2 of Figure 1, following magnetic separation 8 and vibration locker screening 9, dry material char feed 10 is introduced in to an Opposed Jet Classifier Mill 12 (British Rema, UK) via an airstream 11 through an inlet hopper. Entrained char material 10 passes into the body of the mill 12 and descends into a grinding chamber 13 at a lower section thereof. Compressed air at approximately 7 bar is supplied to a series of radially disposed nozzles 15 which are located around the periphery of the grinding chamber 13. Particles of feed char 10 are accelerated to a high velocity by a conveying air stream produced by the nozzles 15 and subjected to high intensity impact/attrition grinding.

Ground material having a lower density and therefore entrained in the expanded airstream ascends to a classifier rotor 17 in an upper section of the mill 12. Classification takes place within a vortex generated by the rotor 17 as a result of the interaction between centripetal and air drag forces acting on the char particles.
Product size is determined by control of the speed of the classifier rotor 17. Particles below a cut-off size of approximately 20μm, and a density of approximately 1g/cm³, pass through the rotor 17 and exit with the entrained air for recovery in a product collection feed 16 which is passed to section 3 of Figure 1. Coarse particles 24 above the cut-off size and which are too dense, on which air drag forces have exceeded the centripetal forces migrate to the walls of the mill 12, and descend into the grinding chamber 13 for further grinding with the incoming feed. High density particles, which are greater than 1g/cm³, are removed from the mill 12 as waste 24. The milling stage takes place in an enclosed environment.

Referring to Section 3 of Figure 1, particles of char 16 produced by the Mill 12, which are less than 20μm in diameter, and also less than 1g/cm³, are sucked out of the mill 12 by an Opposed Jet cyclone/bag 22 for collection.

Referring to Section 4 of Figure 1, the carbon char 19 is fed from the cyclone/bag 22, and is introduced into a mixing tank 26 where it is mixed with demineralised (de-ionised) water 32, which is produced by a water demineraliser 30. This water has had anions and cations removed via ion exchange. Demineralised water 32 is added to the carbon char 19 at a ratio of approximately 1:1 (%wt) although more water could be used to improve efficiency. The char/water mix is then heated to 70°C and continuously mixed with a Cole Palmer mixer 28 to aid the absorption of salts and sulphur into the demineralised water 32, thereby creating a carbon char slurry 34.
The slurry 34 is fed by a pump 36 into a centrifuge 38 (Broadbent Decanter Centrifuge, Thomas Broadbent and Sons Ltd, UK), where it is centrifuged until approximately 10%wt of the water remains in the carbon char slurry 34. The char slurry 34 is then returned back to the mixing tank 26 for further washing with more demineralised water 32 for a second time. In the mixing tank 26, the slurry 34 is again heated to 70°C for approximately 1 hour, and is constantly mixed with the Cole Palmer mixer 28. This second washing step rinses the char slurry 34 again and, following this rinsing process, the char 34 is dewatered in the centrifuge 28 for a second time leaving a high purity carbon black pigment with a moisture concentration of less than 10%wt. The concentration of carbon in the pigment is approximately 90-95% (w/w), and can be used for photocopier toner. The toner can consist of either dry photocopier toner, or liquid toner for use in a laser printer.

Referring to Section 5 in Figure 1, the high purity carbon black having less than 10% retained moisture is then sent to a holding tank 42. The carbon black pigment is now suitable input material for processing into an ink jet ink dispersion which itself can be used to produce an ink jet ink formulation. Firstly, the high purity carbon black pigment is mixed with demineralised water 32 to achieve a total water to carbon black ratio of 4.18:1. The total water is the sum of retained water from Section 4 plus additional demineralised water 32 added thereto. The water and carbon black are constantly mixed in the holding tank 42 with a mixer 28.
Referring to Section 6 of Figure 1, following determination of the batch size to be processed, an ink dispersion is made by mixing 87.7%wt of the carbon black/water with 12%wt of a dispersant 44 (Solsperce 44,000, Avecia Pigments and Additives, Manchester, UK), and 0.3%wt of an anti-foaming agent 46 (Rhodoline DF 6681, Rhodia Industrial Specialities Ltd, UK), in a pre-processing tank 50. The dispersion is centrally stirred for a few seconds with a non-vortexing tube mixer 43. In addition, the tube mixer 43 is moved around the pre-processing tank 50 to eliminate any dead space. Two minutes of mixing per litre of premix in the tank 50 ensures appropriate consistency for pumping into a processing unit 52, which consists of a microniser Buhler ZR 120+ (Buhler Ltd, UK).

The carbon char material particles are less than 20µm in size at this stage and the pre-mix has a consistency to enable the Buhler microniser 52 to achieve an average particle size of 0.060µm to 0.50µm. The microniser 52 is configured by Buhler to ensure compatibility with the input formulation. The pre-mix is fed into the microniser 52 at a constant flow rate of 1 litre per minute, although the flow rate could be between 60L/hr-8000L/hr. The microniser 52 has grinding media, which consists of Yttrium-stabilised Zirconium grinding beads, which are 0.3mm in diameter. The micronising is carried out at the pressure of 0.2 bar, although the pressure could be between 0.1bar-6bar. The machine has a cut-out temperature of 40°C.

Referring to Section 7 in Figure 1, following processing in the Buhler ZR 120+ processing unit 52, the carbon black
dispersion is then fed 87 to a membrane filter 56 which filters out any oversized particles that have resisted milling in the Buhler ZR 120+ processing unit 52. The membrane filter 56 filters out particles greater than 0.50μm in size and these are recycled 58 back to the processing unit 52.

The filtered carbon char dispersion 88 is then sent to a post-processing holding tank 74 and undergoes quality control in a Quality Control area 60 as illustrated in Section 8 of the Figure. As part of ongoing quality control, process data is captured in real-time from all stages of the process 2 and assessed centrally to determine conformity with expectations at each stage of the process 2. The batch can be rejected at any stage if it fails to meet the expected quality parameters and returned for reworking, if necessary. In addition, the finished dispersion held in holding tank 74 is subjected to testing for particle size, appearance on substrate medium, and stability.

Two methods of particle size measurements are used. Firstly, a Malvern Zeta Sizer 62 and, secondly, a high magnification power microscope 64. In addition, stability analyses 66 are carried out involving heating and maintaining the product at a temperature of 70°C for 24 hours.

Following quality control in Section 8, the dispersion 88 is then sent to Section 9 in the Figure, where the dispersion 88 is used for the preparation of an ink jet ink formulation 86. The formulation consists of 50% (w/w) pigment dispersion 88, 10% (w/w) Ethylene glycol 82, 10%
(w/w) Diethylene glycol 84, and 30% (w/w) Deionised water 32.

Referring to Section 10 in Figure 1, the final ink jet ink formulation is then bottled and packaged 68, and then sent to a despatch area 70. Finished carbon black ink jet ink formulation 72 is then ready for distribution.

A second embodiment of the invention is shown in Figures 2, 3 and 4. The second embodiment is a process 2 for refining impure carbon char produced from the pyrolysis of waste tyres. Section 1 and Section 2 of Figure 2 are the same as like sections in Figure 1. Section 3 of Figure 2 is similar to that described in Figure 1 except particles are produced which are less than 5μm in diameter.

The second embodiment as shown in Figures 2 to 4 incorporates a further pyrolysis stage as indicated by Section 4, and a Section 5 which includes an acid wash stage (109) and an alkali wash stage (124).

It is desirable to remove completely, or reduce significantly, the % of metals and silicon still present in the further pyrolysed carbon char. This is undertaken by treating the carbon using acid and alkali respectively. Acid and alkali treatment processes are undertaken using the same equipment and methodology. Therefore, acid and alkali treatments although grouped together in Figure 2, are undertaken in tandem.

In more detail, Figure 2 shows a flow chart of a process for refining impure carbonaceous char in to a dispersion,
which can be used to produce: ink jet printer ink, ultra violet cured ink, wide format ink, and toner for use in laser printers and toner for use in photocopiers.

Waste tyres are initially fed 4 into a pyrolysation unit 6 to produce impure carbon black char 7. The char 7 is then screened using a magnetic screen 8 and a locker screen 9 (Section 1). The magnetic screen 8 removes ferrous material impurities from the carbon char, and the locker screen removes particles in the char which are greater then 1mm in size.

The char particles are then further classified using an opposed jet mill 12, which extracts smaller particles of metal, and grinds the char down such that the average particle size is no greater than 5 microns (Section 2). The char particles are sucked under vacuum out of the Opposed Jet Classifier Mill 12 by a cyclone 22 (Section 3). Char from the Classifier Mill is fed into specially designed stainless steel retorts 100. The retorts (100) are purpose designed to allow the escape of gas generated through pyrolysis of the carbon char feed (99) but also to prevent the egress of air into the retorts (100).

Char filled retorts 100 are then introduced to the secondary pyrolysis furnace 102 (Section 4). The furnace 102 heats the char filled retorts until the temperature at the core of the centre of the retort 100 has reached 800 degrees centigrade. The temperature at the core is maintained through the attachment of thermocouples linked to the furnace control display panel. The core temperature is maintained for five hours then the retorts are allowed to cool within the furnace.
Cooled pyrolysed char filled retorts 104 are transported to Section 5 200 to remove salts and sulphur from the carbon black char. Char is emptied from the retorts into a dissolver vessel 109 containing hydrochloric acid and demineralised water solution drawn from tank 108. The acid slurry 106 is agitated by means of a long shafted stirrer 110. The slurry 106 is heated by steam to boiling and the boiling temperature maintained for one hour.

The slurry is allowed to cool to 60°C with the addition of demineralised water, for handling purposes. The cooled slurry is pumped through a slurry pump 112 into a plate and cloth pigment press 114. The plate and cloth press squeezes out most of the acid solution through discharge pipe 116 leaving a dewatered carbon cake. The resulting cake is then washed in the press using demineralised water until pH neutral.

Section 5 200 further includes a second stage 201 which incorporates a dissolver vessel 120. The acid washed and rinsed char cake is transferred to the second dissolver vessel 120 where it is mixed with a solution of caustic soda and deionised water from tank 122 to form an alkali slurry 124. The slurry is agitated by long shafted stirrer 126 and heated by steam to boiling. The boil temperature is maintained for 1 hour and the alkali char slurry is allowed to cool to 60°C with the addition of demineralised water, for handling and the cooled slurry is pumped through a slurry pump 126 into a plate and cloth pigment press 128. The plate and cloth press 128 squeezes out most of the alkali solution through a discharge pipe.
130. The resulting cake 202 is then washed in the press using demineralised water until pH neutral.

The cake is fed into a holding tank 132 where it is mixed with demineralised water from a header tank 134 a long shafted stirrer is used to maintain agitation Section 6. The high purity slurry is now of sufficiently high carbon content to be suitable for making dispersion.

From the holding tank 132 the high purity slurry is fed into a pre process tank 134 Section 7 and mixed with chemical additives 136. The dispersion slurry 138 is agitated by means of a long shafted stirrer 140. The mix is fed into the Main Processing Machine a Buhler ZR 120 + centrifugal bead mill machine. Milling takes place until particle size of <120 nanometers has been achieved.

The dispersion is fed to post process holding tank 142 Section 8. Samples are taken from the dispersion and analysed at Section 10 for particle size, colour density, viscosity, and surface tension. If the sample passes the quality control it moves to final processing (ink formulation) Section 8. Chemical additives 144 are added to the dispersion in the dosing/mixing tank 146. Ink jet ink has now been produced.

The ink jet ink is pumped through a 500 nanometer filtration process 148 Section 9. Samples are returned to the Quality Control Section 10 for analysis. Ink passing the Quality Control is filled into intermediate bulk carriers 150. Finished ink is stored for orders in the storage bay 152 Section 11. Ink is bottled in Section 12.
using a standard bottling line 154. Bottled ink is transported by courier or post to customers.

An example of the % w/w element impurities remaining in the char following pyrolysis of the carbon char and acid and alkali washing is shown in table 1 below:

<table>
<thead>
<tr>
<th>Elements</th>
<th>Char*</th>
<th>Pyrolysed Char**</th>
<th>**Pyrolysed Char + Acid wash liquor</th>
<th>**Pyrolysed Char + Acid and alkali washing liquor</th>
</tr>
</thead>
<tbody>
<tr>
<td>Na</td>
<td>0.209</td>
<td>&lt;0.03</td>
<td>&lt;0.01</td>
<td>&lt;0.01</td>
</tr>
<tr>
<td>Mg</td>
<td>0.123</td>
<td>0.0868</td>
<td>&lt;0.01</td>
<td>&lt;0.01</td>
</tr>
<tr>
<td>Al</td>
<td>0.205</td>
<td>0.129</td>
<td>&lt;0.01</td>
<td>&lt;0.01</td>
</tr>
<tr>
<td>Si</td>
<td>3.5</td>
<td>2.6</td>
<td>2.6</td>
<td>0.01</td>
</tr>
<tr>
<td>S</td>
<td>3.9</td>
<td>1.0</td>
<td>0.01</td>
<td>0.01</td>
</tr>
<tr>
<td>Cl</td>
<td>0.099</td>
<td>0.0445</td>
<td>&lt;0.01</td>
<td>&lt;0.01</td>
</tr>
<tr>
<td>K</td>
<td>0.104</td>
<td>0.0899</td>
<td>&lt;0.01</td>
<td>&lt;0.01</td>
</tr>
<tr>
<td>Ca</td>
<td>1.0</td>
<td>0.802</td>
<td>&lt;0.01</td>
<td>&lt;0.01</td>
</tr>
<tr>
<td>Ti</td>
<td>0.0291</td>
<td>0.0226</td>
<td>0.0226</td>
<td>0.0226</td>
</tr>
<tr>
<td>Fe</td>
<td>1.0</td>
<td>0.715</td>
<td>&lt;0.01</td>
<td>&lt;0.01</td>
</tr>
<tr>
<td>Co</td>
<td>0.0295</td>
<td>0.0257</td>
<td>0.0257</td>
<td>0.0257</td>
</tr>
<tr>
<td>Cu</td>
<td>0.0696</td>
<td>0.0513</td>
<td>&lt;0.01</td>
<td>&lt;0.01</td>
</tr>
<tr>
<td>Zn</td>
<td>3.5</td>
<td>2.94</td>
<td>0.01</td>
<td>0.01</td>
</tr>
<tr>
<td>Br</td>
<td>0.0679</td>
<td>0.0404</td>
<td>0.01</td>
<td>0.01</td>
</tr>
<tr>
<td>C</td>
<td>85.8</td>
<td>91.38</td>
<td>97.25</td>
<td>99.832</td>
</tr>
</tbody>
</table>

The carbon content is shown to be 99.8% as determined by summing the impurity total and subtracting from 100.

All analysis was done on dry samples.
Char* - This is the carbon black char produced from the pyrolyisation of scrap tyres. The resultant char is magnetically separated using density and particle size separation. The particle size is less than 5 microns.

**Pyrolysed char- this is char* that has been pyrolysed to 800 centigrade.

Pyrolysis of the carbon char feed derived from the pyrolyisation of scarp tyres reduces the levels of impurities. The carbon content in the char is raised from approximately 85% to 90% through further pyrolysis. The most notable improvement is Sulphur reduction. Sulphur content is reduced from approximately 4% to 1% after further pyrolysis.

As a result of the further pyrolysis of the carbon char feed, the carbon level rises to over approximately 90%. But this is still too low to be suitable for high grade ink jet ink. Experience teaches that the higher the carbon purity the finer the ink.

Experience teaches that using the raw feed to create the dispersion results in a viscous dispersion. High viscosity is undesirable and is carried from dispersion into the ink. Ink formulated from a viscous dispersion does not print properly. High viscosity is due to the presence of part pyrolysed and unpyrolysed polymeric material contained in the tyre matrix and remaining in the raw char after initial pyrolysis. The presence of hydrocarbons in raw char is evidenced by toluene tests (toluene shows brown) after secondary pyrolysis toluene test shows clear.
Unexpectedly, secondary or further pyrolysis of the carbon black char has also resulted in both cost and quality improvements. The wettability of the pyrolysed carbon black char is improved, significantly reducing the requirement for expensive wetting dispersants. Further, due to reduced surfactant at the dispersion stage, ink formulated from the dispersion is less viscous: it therefore prints better. Also reduced surfactant provides significant improvement in colour density at the finished ink stage.

Therefore, there are unexpected cost and quality benefits associated with pyrolysis of the carbon black char which combine with the anticipated benefits of raised carbon content and a reduction in the problem of high viscosity.

Advantages of the refining method of using post pyrolysis tyre char as defined in the first embodiment, or the method of using post pyrolysis tyre char which is then further pyrolysed as defined in the second embodiment, over virgin cracked carbon black are that it helps to solve the problem of what to do with scrap tyres in line with EU Directive. Also, it is an effective use a waste char material that would otherwise be sent to landfill. The method does not use additional virgin cracked carbon black and also saves a lot of oil (at ratio of about 6 tonnes of oil saved per tonne of carbon black produced). The method also has low carbon dioxide emissions (at about 3 tonnes of carbon dioxide per tonne of oil saved, thereby reducing 18 tonnes of carbon dioxide emissions per tonne of virgin carbon black replaced). Also, the method
produces an agglomeration resistant material, be it either the pigment, formulation or the dispersion.

Advantages of the method described in the first embodiment of post-pyrolysis tyre char over other carbon char purification methods are that it uses mechanical and not chemical separation methods to raise carbon purity. In addition, the method uses no acid/alkali or solvents, but uses a variable magnetic field to remove impurities such as steel wire. The method removes particles larger than 1mm from the feed as these contain greater impurities than smaller particles. The method removes high density particles, as above these contain higher levels of impurity. The pigment produced by the claimed method has applications as an ink dispersion ink jet printer ink and also as a photocopier and laser printer toner.

The advantage of the method described in the second embodiment which incorporates the pyrolysis of the carbon char feed, together with an acid wash and alkali wash, is it ensures that the resultant char is of a high quality. The carbon black char produced is generally free from short chain hydrocarbons and aromatics which ultimately cause blocking of the ink cartridge. Further, without the pyrolysisation of the carbon black char, a large amount of dispersant would be necessary to “wet” the carbon. The addition of dispersant results in a low colour strength of the carbon black product.

Advantages of the finished ink jet printer ink described herein are that it has an improved quality, and an increased colour density, i.e. the ink produced is a blacker black. In addition, the ink has a high resistance
to bleed, i.e. the black does not bleed into other colours when jetted onto a substrate. The ink is resistant to "feather", i.e. the ink produces sharp images and lines, which are not feathery. The ink has a high stability, and is resistant to agglomeration therefore particles do not "clump" or join together. Hence, there is a low probability of blocking printer nozzles and orifices. Furthermore, solids remain in suspension, i.e. they do not separate from the liquid carrier as quickly as many other inks increasing shelf life.

Advantages of the dispersion described herein over other ink dispersions are that it is aqueous based and no solvents have been used during its manufacture. Also, the dispersion is re-agglomeration resistant.

The reader's attention is directed to all papers and documents which are filed concurrently with or previous to this specification in connection with this application and which are open to public inspection with this specification, and the contents of all such papers and documents are incorporated herein by reference.

All of the features disclosed in this specification (including any accompanying claims, abstract and drawings), and/or all of the steps of any method or process so disclosed, may be combined in any combination, except combinations where at least some of such features and/or steps are mutually exclusive.

Each feature disclosed in this specification (including any accompanying claims, abstract and drawings), may be replaced by alternative features serving the same,
equivalent or similar purpose, unless expressly stated otherwise. Thus, unless expressly stated otherwise, each feature disclosed is one example only of a generic series of equivalent or similar features.

The invention is not restricted to the details of the foregoing embodiment(s). The invention extends to any novel one, or any novel combination, of the features disclosed in this specification (including any accompanying claims, abstract and drawings), or to any novel one, or any novel combination, of the steps of any method or process so disclosed.
CLAIMS

1. A method of refining carbon black char derived from the pyrolysation of scrap tyres, comprising a pyrolysation step wherein the carbon black char is pyrolised to produce a pyrolysed carbon black char feed.

2. A method as claimed in claim 1, wherein the pyrolysation step occurs after the steps of
   a) screening the carbon black char derived from pyrolysation of scrap tyres;
   b) magnetic separation
   c) size classification
   d) density classification.

3. A method as claimed in claims 1 or 2, wherein the pyrolysation step occurs in a chamber having a one-way valve.

4. A method as claimed in any one of the preceding claims, wherein the pyrolysation step occurs at a temperature of 800°C.

5. A method as claimed in any one of the preceding claims, wherein the method comprises a first washing step.

6. A method as claimed in claim 5, wherein the first washing step comprises washing the pyrolysed carbon black char feed with a first washing liquor.
7. A method as claimed in claim 6, wherein the first washing liquor is a hydrochloric acid solution.

8. A method as claimed in any one of the preceding claims, wherein the method comprises a second washing step.

9. A method as claimed in claim 8, wherein the second washing step comprises washing the pyrolysed carbon black char feed with a second washing liquor.

10. A method as claimed in claim 9, wherein the second washing liquor is an alkaline solution.

11. A method of refining carbon black char comprising the steps of:

   a) Pyrolysation of scrap tyres to produce an impure carbon black char feed;
   b) classification of the carbon black char feed;
   c) pyrolysation of the carbon black char feed;
   d) acid washing of the carbon black char feed;
   e) alkali washing of the carbon black char feed;
   f) demineralised water washing of the carbon black char feed; and
   g) processing of the carbon black char feed to produce a carbon black product.

12. A method as claimed in claim 11, wherein step c) occurs before step d).

13. A method as claimed in claim 12, wherein step step c) occurs after step b).
14. A method as claimed in any one of claims 11 to 13, wherein step g) comprises the steps of milling and dispersion of the carbon black char feed.

15. A method as claimed in any one of claims 11 to 14, wherein step b) removes particles greater than 5µm.

16. A method of producing a pigment comprising carbon black, the method comprising a pyrolyisation step wherein the carbon black char is pyrolysed to produce a pyrolysed carbon black char feed.

17. A method as claimed in claim 16, wherein the method comprises washing the pyrolysed carbon black char feed with a first washing liquor.

18. A method as claimed in claim 17, wherein the first washing liquor is an acidic solution.

19. A method as claimed in any one of claims 16 to 18, wherein the method comprises washing the pyrolysed carbon black char feed with a second washing liquor.

20. A method as claimed in claim 19, wherein the second washing liquor is an alkaline solution.

21. A method as claimed in any one of claims 16 to 20, wherein particles greater than 5µm in size are removed from the carbon black char feed.

22. A pigment comprising carbon black wherein the pigment is derived from a feed of pyrolysed carbon black char.
23. A pigment as claimed in claim 22, wherein the pyrolysed carbon black char is washed with at least a first washing liquor.

24. A composition comprising anti-foaming agent, surfactant, and carbon black, wherein the carbon black is derived from pyrolysed carbon black char produced from the pyrolysis of scrap tyres.

25. A method of refining carbon black char as claimed in any one of the preceding claims, the method comprising removing particles greater than approximately 1mm in size from a carbon black char feed.

26. A method as claimed in any one of the preceding claims, wherein the removal of particles greater than 1mm in size produces a pigment comprising carbon.

27. A method as claimed in claim 26, wherein the pigment comprises at least 50% (w/w) carbon.

28. A method as claimed in any preceding claim, wherein the method comprises classifying the carbon char feed by size such that at least 50%(w/w) of the feed has a particle size of less than approximately 1mm.

29. A method as claimed in any preceding claim, wherein the particles greater than 1mm are removed from the carbon char feed by screening.

30. A method as claimed in any preceding claim, wherein the method further comprises a step of magnetically separating impurities from the carbon black char feed.
31. A method as claimed in claim 30, wherein the method comprises varying a magnetic field in the vicinity of the carbon char feed to which the magnetic impurities are attracted, thereby separating the magnetic impurities therefrom.

32. A method as claimed in any preceding claim, wherein the carbon black char feed comprises a dry powder.

33. A method as claimed in any preceding claim, wherein the method comprises a second size classification step.

34. A method as claimed in any one of the preceding claims, wherein the method comprises milling and/or grinding the feed.

36. A method as claimed in any one of the preceding claims, wherein the second size classification step removes particles from the feed so that at least 80% (w/w) of the feed has a particle size of less than approximately 5µm.

37. A method as claimed in any preceding claim, wherein the method comprises classifying the carbon char feed by density.

38. A method as claimed in any preceding claim, wherein the method comprises removing particles from the feed which have a density greater than approximately 0.5g/cm³.
39. A method as claimed in any preceding claim, wherein the method comprises removing sulphur compounds from the impure carbon black char feed.

40. A pigment comprising carbon black derived from a feed of carbon black char, wherein particles greater than approximately 1mm in size are removed from the carbon black char feed.

41. Apparatus for refining carbon black char, the apparatus comprising means to remove particles greater than approximately 1mm in size from a carbon black char feed.

42. Apparatus as claimed in claim 41, wherein the apparatus comprises washing means adapted to substantially remove sulphur and, metallic salts, from the feed.

43. A composition comprising anti-foaming agent, surfactant, and carbon black, wherein the carbon black is derived from a feed of carbon black char, wherein particles greater than approximately 1mm in size are removed from the carbon black char feed.

44. A composition as claimed in claim 43, wherein the surfactant comprises a dispersant.

45. An ink formulation comprising the composition according to any one of the preceding claims, ethylene glycol, and diethylene glycol.
55. An ink formulation as claimed in claim 45, wherein the ink formulation is used for the manufacture of an ink jet printer ink.

56. A method of refining carbon black char substantially as herein described with reference to, and as illustrated, in the accompanying drawings.
ACID WASH

AGITATE

HEAT

COOL

PRESS

DEMINERALISED WATER WASH

CAKE

FIG. 3

SUBSTITUTE SHEET (RULE 26)
CHAR FEED
   ↓
SCREENING
   ↓
CLASSIFICATION
   ↓
CYCLONIC SEPARATION
   ↓
PYROLYSATION
   ↓
ACID WASH
   ↓
ALKALI WASH
   ↓
DEMINERALISED WATER WASH
   ↓
PREPROCESSING
   ↓
MILLING
   ↓
FINAL PROCESSING
   ↓
INK JET INK

FIG. 5