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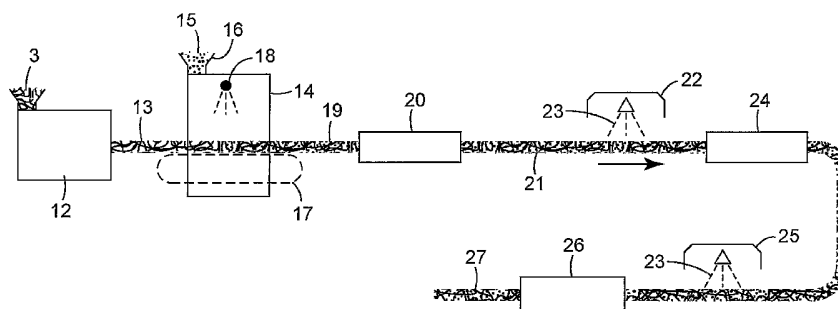
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(54) Title: METHOD OF MANUFACTURING NONWOVEN ABRASIVE ARTICLES USING DRY PARTICULATE MATERIAL



(57) Abstract: A method of making a nonwoven abrasive article (17) includes the steps of forming a three-dimensional nonwoven fibrous web (3) contacted with dry particulate material that includes fusible binder particles (5); exposing the web to conditions that cause the binder particles to form a flowable liquid binder, and then solidifying the liquid binder to form bonds between the fibres of the web and thereby provide a pre-bonded web (11); and applying abrasive particles to the pre-bonded web, and bonding the abrasive particles to the fibres of the pre-bonded web to provide the abrasive article. The fusible binder particles (5) can be applied to the nonwoven fibrous web (3) after the latter has been formed, for example by spraying the binder particles at the web using an electrostatic powder spray gun (8). The abrasive particles may be applied to the pre-bonded web in a slurry (13) formed by mixing the particles with a make-coat binder resin that is subsequently cured to bonded the abrasive particles to the fibres of the pre-bonded web.

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For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

METHOD OF MANUFACTURING NONWOVEN ABRASIVE ARTICLES USING DRY PARTICULATE MATERIAL

The present invention relates to the manufacture of nonwoven abrasive articles.

5

Nonwoven abrasive articles comprising a nonwoven fibrous web to which abrasive particles are attached are known and are used extensively for cleaning, abrading, finishing and polishing applications on a variety of surfaces. Examples of such nonwoven abrasive articles, and methods by which they may be manufactured, are described in US-A-2 958 593. One method comprises forming an open, air-laid fibrous web, and spraying one surface of the web with a resin-abrasive slurry which is then cured. The other surface of the web is then sprayed with the same resin-abrasive slurry, which is also cured. Roll-coating and dip-coating are mentioned in US-A-2 958 593 as alternative methods of applying the resin-abrasive slurry, and it is also mentioned that the resin and the abrasive particles may be applied separately. It is further stated that it may be desirable to employ one type of binder (for example, a light treatment of an elastomer) for adhesively bonding the web fibres together to integrate and unify the web, and a grit binder of different composition for bonding the mineral grains to the integrated and unified web.

20

Other nonwoven abrasive articles and methods for their manufacture are described in US-A-2 327 199; 2 375 585; 3 175 331; 4 227 350; 4 355 489; 4 486 200; 4 991 362; and 5 363 604

25 US-A-5 681 361 describes a method of making nonwoven abrasive articles, in which binder material and abrasive particles are applied to a nonwoven web in the form of dry particulate material, to reduce the use of liquid organic solvents during the manufacturing process and thereby address environmental concerns associated with the emission of volatile organic compounds (VOCs). Optionally, the fibres of the web are pre-bonded together at their mutual points of contact before the abrasive particles and the particulate binder material are applied. That pre-bonding may be effected either (i) 30 by applying, to the web, a liquid pre-bond resin that is then cured, or (ii) by including,

within the construction of the web, melt-bondable fibres that are caused to melt and then allowed to re-solidify.

The present invention is also concerned with the environmental aspects of the manufacture of nonwoven abrasives (including the use of liquid organic solvents and the accompanying risk that VOCs will escape into the atmosphere, the energy requirements of the manufacturing process, and the waste produced), and is additionally concerned with enabling nonwoven abrasive articles to be manufactured from alternative materials not previously considered suitable for that purpose.

10

The present invention provides a method of making an abrasive article, including the steps of

(i) forming a three-dimensional nonwoven fibrous web contacted with dry particulate material that includes fusible binder particles;

15

(ii) exposing the web to conditions that cause the binder particles to form a flowable liquid binder, and then solidifying the liquid binder to form bonds between the fibres of the web and thereby provide a pre-bonded web; and

(iii) applying abrasive particles to the pre-bonded web, and bonding the abrasive particles to the fibres of the pre-bonded web to provide the abrasive article.

20

In a method in accordance with the invention, a pre-bonded web is produced using dry materials only: consequently, no VOCs need be emitted during this part of the method, and the energy requirements are less than if liquid pre-bond resins were used. Any excess binder particles applied to the web need not be wasted but can be recovered and re-used. During the pre-bonding stage, an open, lofty web having adequate structural strength for use in a nonwoven abrasive article can be produced using fewer, or less resilient, fibres than would be required if the web were roll-coated or sprayed with liquid pre-bond resins.

25

30 The terms "open" and "lofty" indicate that the pre-bonded web is of comparatively low density, having a network of many, relatively large, intercommunicated voids that comprise the greater amount (more than 50%, preferably substantially more than 50%)

of the volume occupied by the web. In the context of the present invention, the terms indicate that the bonded web has a density no greater than 50 kg/m^3 , preferably no greater than 30 kg/m^3 (especially when the web comprises synthetic fibres).

5 By way of example only, methods of manufacturing nonwoven abrasive materials in accordance with the present invention will now be described.

A first method in accordance with the invention comprises the steps of:

- (i) (a) forming fibres into a three-dimensional nonwoven web and then
- 10 (b) contacting the web with dry particulate material that includes fusible binder particles;
- (ii) exposing the web to conditions that cause the binder particles to form a flowable liquid binder, and then solidifying the liquid binder to form bonds between the fibres of the web and thereby provide a pre-bonded web; and
- 15 (iii) applying abrasive particles to the pre-bonded web, and bonding the abrasive particles to the fibres of the pre-bonded web to provide the abrasive article.

Each of those steps will now be described in greater detail.

Step (i)(a) Forming fibres into an open, lofty, three-dimensional nonwoven web

20 The fibers that can be used to form the nonwoven web include both natural and synthetic fibers and mixtures thereof. The fibers are typically in the form of staple and may be tensilized. The fibers can be curled, crimped or straight. Suitable synthetic fibers include those made of polyester (e.g., polyethylene terephthalate), polyamide (e.g., hexamethylene adipamide, polycaprolactum and aramids), polypropylene, acrylic

25 (formed from a polymer of acrylonitrile), rayon, cellulose acetate, polyvinylidene chloride-vinyl chloride copolymers, and vinyl chloride-acrylonitrile copolymers, as well as carbon fibres and glass fibres. Suitable natural fibers include those of cotton, wool, jute, coco, sisal, flax and hemp. An important consideration in the selection of the fiber is that it is capable of withstanding the process temperatures to which it will

30 subsequently be exposed (see below). The fibers used may be virgin fibers or waste fibers reclaimed from garment cuttings, carpet manufacturing, fiber manufacturing, or textile processing, and so forth.

The fineness or linear density of the fibers used may vary widely, depending upon the results desired. Coarse fibers are generally more conducive to making abrasive articles for rough abrading jobs, while finer fibers are generally more appropriate for less aggressive scouring applications. Useful fibers generally are those having a linear density of from about 6 to 300 denier (more especially 10 to 200 denier) although finer or coarser fibers (or mixtures thereof) may be used depending, for example, on the application envisaged for the finished abrasive article.

10 If fibres are provided in the form of bales, the bales should be opened before use. The selected fibres can then be dry-laid into an open, lofty, three-dimensional nonwoven fibrous web in any suitable way. The web may, for example, be air-laid or carded and cross-lapped, and may additionally be needle-tacked. Processes for producing such webs are well known. A preferred type of nonwoven web is an air-laid web as
15 described in US-A-2 958 593.

An air-laid nonwoven web can be formed by a "Rando-Webber" machine commercially available from Rando Machine Co., Macedon, N.Y. With such processing equipment, a range of fibre lengths can be used although an average fiber length of about 10 cm. is most usual. However, with this, as with other types of conventional web forming equipment, fibers of different lengths, or combinations thereof also can be utilized to form the nonwoven web. As already mentioned, there are no particular limitations on the thickness of the fibers (apart from those imposed by processing considerations), as long as due regard is given to the resilience and toughness ultimately desired in the
25 resulting web.

The physical characteristics of the web at this stage should be selected having regard to the desired properties of the abrasive article that is to be produced. In some cases a very open, low density, web might be required (for example, when the abrasive article is a domestic scouring pad): in such a case, an air-laid web produced using fine fibres could
30 be preferred. In other cases a less open, higher density, web might be required: in such a case, a carded, cross-lapped and needle-tacked web using coarser fibres could be

preferred. In each case, the thickness of the web will depend on the way in which the web is produced and also on the nature of the fibres and the quantity used per unit area.

Step (i)(b) Contacting the web with dry particulate material that includes fusible binder particles

5 The nonwoven web that is formed as described in step (i)(a) above is then contacted with dry particulate material that includes fusible binder particles. The binder will subsequently be activated, as described below, to form bonds between the fibres of the web and thus provide a pre-bonded web to which abrasive particles are subsequently
10 applied. The binder particles should therefore be selected having regard to the nature of the web fibres and the subsequent processing steps to which the pre-bonded web will be subjected, and having regard also to the desired properties of the abrasive article that is to be produced.

15 Particulate materials suitable for use in bonding nonwoven webs are known, and include thermosetting and thermoplastic powders that are activated by heat, as well as powders that are activated in other ways (for example, by moisture). Particulate materials suitable for bonding nonwoven webs for various purposes are described, for example, in US-A-4 053 674, 4 457 793, 5 668 216, 5 886 121, 5 804 005, 5 9767 244,
20 6 039 821, 6 296 795, 6 458 299, and 6 472 462. The particulate binder materials most suitable for use in the manufacture of abrasive articles are those that will provide an abrasive article with good strength and water/heat resistance and, for use with a nonwoven web as described above, the particulate binder materials should be capable of being activated without damaging the web fibres.

25 Thermosetting particulate binder materials that have been proposed for use in the manufacture of nonwoven abrasive articles include formaldehyde-containing resins, such as phenol formaldehyde, novolac phenolics and especially those with added crosslinking agent (e.g., hexamethylenetetramine), phenoplasts, and aminoplasts;
30 unsaturated polyester resins; vinyl ester resins; alkyd resins, allyl resins; furan resins; epoxies; polyurethanes; polyimides; and thermosetting acrylates.

Thermoplastic particulate binder materials that have been proposed for use in the manufacture of nonwoven abrasive articles include polyolefin resins such as polyethylene and polypropylene; polyester and copolyester resins; vinyl resins such as poly(vinyl chloride) and vinyl chloride-vinyl acetate copolymers; polyvinyl butyral; cellulose acetate; acrylic resins including polyacrylic and acrylic copolymers such as acrylonitrile-styrene copolymers; polyamides (e.g., hexamethylene adipamide, polycaprolactum), and copolyamides.

Mixtures of the above thermosetting and thermoplastic particulate binder materials may also be used.

The methods and equipment useful for applying the particulate binder material to the nonwoven web may be selected from among any known to be suitable for achieving an even distribution of the required amount of binder material throughout the web.

Suitable methods include powder scattering or drop coating and powder spraying or sifting. In some cases, the application of particulate binder material to a nonwoven web may be followed by an impregnation step in which the web is passed through an alternating electrical field to distribute the binder material throughout the thickness of the web. A preferred method of applying the particulate binder material to the nonwoven web uses an electrostatic powder spray gun, of the type known for use in powder coating applications, to direct electrically-charged binder particles downwards towards the web while the latter is being carried on a grounded, electrically-conductive open-mesh conveyor. The binder particles will penetrate the whole thickness of the web, under the combined effects of electrostatic attraction, flow of atomising air from the spraygun, and gravity; and excess binder material that passes through the web can be collected below the conveyor and recycled. In some cases, the web can be pre-moistened to increase the amount of binder particles retained within it.

The size of the binder particles is selected to suit the method by which the binder material is applied to the nonwoven web and to ensure that the particles are small enough to penetrate into the interstitial spaces between the fibers of the web. In general,

the particle size of the binder particles is less than about 1 mm, preferably less than about 500 micrometers. In the case in which the binder particles are to be applied to the web using an electrostatic powder spray gun, they preferably have a particle size no greater than 200 micrometers. To minimize wastage, the amount of binder material applied to the nonwoven web should be adjusted to the minimum amount consistent with providing adequate bonding of the web having regard to the subsequent processing to which it will be subjected, and the nature of the abrasive article for which the bonded web will be used.

10 The binder material may be applied to the web in admixture with non-resinous powdered substances such as fillers, catalyst accelerators, flow aids, pigment powders, grinding aids, and anti-smearing agents.

Step (ii) Bonding the web fibres

15 The nonwoven web containing the binder particles, formed as described in steps (i)(a) and (b) above, is then exposed to conditions that will cause the binder particles to form a flowable liquid. It is found that the binder liquid tends to flow along and coat the web fibres so that, when the binder liquid subsequently solidifies, the web fibres will be bonded together at their mutual contact points.

20

In the case, for example, in which the binder material is a heat-activated thermosetting powder (for example, a powdered epoxy resin), the nonwoven web containing the binder material is heated to at least the cure temperature of the resin. To prevent heat damage or distortion to the web, the cure temperature of the resin should be below the melting point (where applicable) of the web fibers. The first effect of a temperature rise sufficiently above the glass transition temperature of the resin will be a softening of the binder material into a flowable fluid-like state, which will allow the resin to wet or contact the surface of the web fibres. Prolonged exposure to a sufficiently high temperature will then trigger a chemical reaction in the resin, forming a cross-linked three-dimensional molecular network that corresponds to a rigid plastic and binds the web fibres together at their mutual contact points.

30

In the case, for example, in which the binder material comprises semi-crystalline thermoplastic particles (for example, polyolefins, hexamethylene adipamide, polycaprolactum), it is preferred to heat the nonwoven web containing the binder material to at least the melting point of the binder material whereupon the thermoplastic particles become molten and form a flowable fluid. To prevent heat damage or distortion to the web, the melting point of the binder material should be below the melting point (where applicable) of the web fibers (or brought into this range by incorporation of a plasticizer). Where non-crystalline thermoplastic particles are used as the binder material (e.g., vinyl resins, acrylic resins), the nonwoven web containing the binder material is heated above the glass transition and rubbery region points of the binder material until the fluid flow region is achieved. In both cases, after a flowable binder fluid has been produced, the nonwoven web is allowed to cool so that the binder fluid solidifies and binds the web fibres together at their mutual contact points.

In the case, for example, of a moisture-activated binder material (for example, a material based on a vinyl ester copolymer), the nonwoven web containing the particulate binder material is sprayed with water to produce a flowable binder fluid. Thereafter, the web is heated to a temperature sufficient to remove the moisture without damaging the fibres and thus leave the fibres bound together at their mutual contact points.

It may be desired that the pre-bonded web at this stage has a certain minimum thickness. For example, when the web comprises natural fibres and is to be used to form domestic scouring pads, a minimum thickness of 5 mm may be preferred.

25

Step (iii) Applying and bonding abrasive particles to the pre-bonded web

Any appropriate method can be used to apply and bond abrasive particle to a pre-bonded web produced as described in step (ii) above, having regard to the nature of the pre-bonded web and the abrasive article that is to be produced.

30

Abrasive particles suitable for application to a pre-bonded web include all known abrasive particulate materials as well as combinations and agglomerates of such

materials. The abrasive particles may be of any size, from less than one micrometer to 2 mm or greater. Included among the suitable abrasive materials are particles of inorganic materials, for example aluminum oxide including ceramic aluminum oxide, heat-treated aluminum oxide and white-fused aluminum oxide; silicon carbide; tungsten carbide; alumina zirconia; diamond; ceria; cubic boron nitride; silicon nitride; garnet; and combinations of the foregoing. It is contemplated that abrasive agglomerates may also be used, such as those described in U.S. Pat. Nos. 4,652,275 and 4,799,939. Suitable abrasive particles also include softer, less aggressive materials such as thermosetting or thermoplastic polymer particles as well as crushed natural products such as crushed nut shells, for example. Suitable polymeric materials for the abrasive particles include polyamide, polyester, poly(vinyl chloride), poly(methacrylic) acid, polymethylmethacrylate, polycarbonate, polystyrene and melamine-formaldehyde condensates. The selection of particle composition and particle size will depend on the contemplated end use of the finished abrasive article, taking into account the nature of the workpiece surface to be treated by the article and the abrasive effect desired. The abrasive particles should have a particle size small enough to allow penetration of the particles into the interstices of the pre-bonded nonwoven web. Chemically-active particles may also be used in combination with the aforementioned abrasive particles, including particles known to be effective as grinding aids such as those comprising poly(vinyl chloride) as well as particles providing effective lubricating properties in the finished article such as those comprising stearates of lithium and zinc, stearic acid and the like.

The abrasive particles may be mixed with a liquid binder composition to form a slurry which is then applied to the pre-bonded nonwoven web in any suitable way (for example, by spraying or by roll-coating). The slurry may be applied to one side only of the nonwoven web or to both sides either simultaneously or in turn. The binder composition is then cured to bind the abrasive particles to the fibres of the web.

As an alternative, the liquid binder composition may be applied alone to the pre-bonded web (for example, by spraying or by roll-coating) following which the abrasive particles can be drop coated, sprinkled, sprayed, or the like, in a dry condition upon a

surface of the web, for example by conveying the web beneath an abrasive particle dispenser. The binder composition is then cured to bind the abrasive particles to the fibres of the web.

- 5 As a further alternative, the abrasive particles may be blended with a powdered resin binder, the blend then being applied in dry form to the pre-bonded nonwoven web.

The binder used to bond the abrasive particles to the pre-bonded nonwoven web can be any resin or adhesive known to be suitable for use as a make coat in abrasives
10 manufacture, including water-based resins. Examples of make coat compositions suitable for use in the manufacture of nonwoven abrasive materials are described in US-A-5 591 239, US-A-5 919 549, EP-A-0 776 733 and WO 01/62442. Preferred binders include phenolic resins (more especially for harder-wearing abrasive articles) and latex resins (more especially for softer abrasive articles, e.g. non-scratch for
15 bathroom cleaning), and they may contain additives such as fillers, lubricants, grinding aids, wetting agents or surfactants, defoaming agents, and pigments or dyes.

If desired, a further resin layer may be applied to the web after the abrasive particles have been attached. This optional resin layer (also known as a size coat) will serve to
20 consolidate the nonwoven abrasive product and increase its wear resistance.

Description with reference to the drawings

Exemplary methods of the invention, in accordance with the above, will now be described with reference to the accompanying drawings, in which:

- 25 Fig. 1 is a schematic illustration of a method of making an abrasive article; and Fig. 2 illustrates a modification of part of the method of Fig. 1.

In the method illustrated in Fig. 1, fibres 1 are supplied to web-forming equipment 2, for example a "Rando Webber" machine, to produce a nonwoven fibrous web 3 that is
30 fed into a powder-coating booth 4 where it is contacted by a particulate pre-bond resin 5 supplied from a fluidizing hopper 6. Optional dry particle additives (such as pigment powder and flow aids) that are to be applied to the nonwoven web at this stage may be

premixed with the pre-bond resin 5 in the hopper 6. The nonwoven web 3 is conveyed through the powder-coating booth 4 on a metallic open mesh conveyor belt 7 that is electrically-grounded, and the particulate resin is directed at the web from above by an electrostatic powder spray gun 8. Any resin that passes through the web 3 and the conveyor belt 7 is collected at the bottom of the booth 4 and can be re-used.

If desired, the web 3 can now be turned over and conveyed for a second time through the powder coating booth 4 to increase the amount of resin powder 5 that is loaded into the web at this stage.

10

The powder-containing nonwoven web 9 is then exposed to conditions that will liquefy the resin particles to a flowable condition, following which the resin is cured to form bonds between the web fibres. In this example, the web is passed through an oven 10 in which it is heated first to liquefy the resin so that it will coat the web fibres, and then to cure the resin so that it will bond the fibres together at their mutual contact points. The pre-bonded web 11 leaves the oven 10 and is allowed to cool.

15

The pre-bonded web 11 is then passed through a first spray booth 12 in which one surface of the web is sprayed with a slurry 13 of abrasive particles mixed with a liquid make-coat binder resin which is subsequently cured by passing the web through an oven 14. The web then passes through a second spray booth 15 in which the other surface of the web is sprayed with the same abrasive-resin slurry 13. The second resin coating is then cured in a second oven 16.

20

In an alternative to the arrangement just described, the second spray booth 15 and the second oven 16 are omitted and, instead, the web 11 is turned over when it has left the oven 14 and is conveyed again through the spray booth 12 so that the other side of the web can be sprayed with the slurry 13. The web is then passed for a second time through the oven 14.

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In either case, the resulting nonwoven abrasive web 17 can then be converted (following storage if required) into abrasive articles such as, for example, abrasive pads

for hand use or domestic scouring pads as described in our corresponding patent application of even date (applicant's ref. no. 57906GB002).

In a modified version of this method, the particulate pre-bond resin 5 may be mixed with the web fibres 1 prior to the formation of the nonwoven web in the web-forming equipment 2. In that case, the powder-coating booth 4 is omitted. In yet another modified version, the powder-coating booth 4 may be replaced by the equipment illustrated in Fig. 2, comprising a powder scattering unit 20 and a powder impregnation unit 21. In that case, the web 3 from the web-forming equipment 2 passes into the unit 20, where the particulate pre-bond resin 5 (together with any optional dry particle additives) is distributed evenly from a dispenser 22 over the upper surface of the web. Any resin that happens to pass through the web is collected at the bottom of the unit 20 and can be re-used. The web then passes into the impregnation unit 21, where it passes between two electrode plates 23 across which an alternating voltage is applied: the effect of this is to distribute the resin powder 5 throughout the thickness of the web, following which the web passes to the oven 10 as in Fig. 1. Brushes 24, contacting the upper and lower surfaces of the web are located downstream of the impregnation unit 21 to remove any excess resin powder, which can be collected and re-used.

A method of the type illustrated in Fig. 2 is described in EP-A-0 914 916, while a further alternative method of contacting a fibrous web with a powder is described in EP 0 025 543.

Examples

Embodiments of the invention are described in the following non-limiting examples, in which all the parts and percentages are by weight unless indicated otherwise.

The examples used the following materials, equipment and test methods:

Materials

Epoxy resin powder: "Beckrypox AF4" low temperature cure black thermoset powder (mean particle size 35 microns) from Dupont of Montbrison, France.

Copolyamide resin powder: "Vestamelt 350 P1" thermoplastic powder 0-80 microns from Degussa of Marl, Germany.

Powder flow aid: "Aerosil 200" hydrophilic fumed silica powder from Degussa of Marl, Germany.

- 5 **Nylon 20 dtex staple fiber:** medium tenacity nylon 6.6 staple fiber type "RT174MT" from Rhodia of Neumunster, Germany.

Nylon 80 dtex staple fiber: nylon 6.6 staple fiber type "R130" from Rhodia of Arras, France.

Sisal fibre: cut fibre from Caruso of Ebersdorf, Germany.

- 10 **Coco fibre:** cut fibre from Caruso of Ebersdorf, Germany.

Poly(vinyl chloride) particles: "Etinox 631" from Aiscondel, Spain.

Corundum particles: very fine grade (average particle size approximately 50 microns) brown fused aluminium oxide from Pechiney, France.

Latex resin: "Styrofan ED609" from BASF, Spain.

- 15 **Cross-linking agents:** (i) Cymel 303 and (ii) Cymel 307 from Dyno Cytec, Norway.

Phenolic resin: "7983SW" from Bakelite AG of Iserlohn-Letmathe, Germany.

Filler: calcium carbonate.

Pigment: red or black, as required.

20 **Equipment**

Fiber opener: from Laroche of Cours La Ville, France

"Rando Webber": an air-lay nonwoven web forming machine from Rando Machine Co. of Macedon, NY, USA

- 25 **Web humidifier:** a water spray head of a type used for room humidification, available from Hydrofog of Chanteloup les Vignes, France.

- Powder coating equipment:** "Versaspray II" electrostatic spray gun(s) from Nordson of Westlake, Ohio, USA, installed in a powder coating booth (also available from Nordson) and directed downwards towards a 30cm wide horizontal metallic open mesh conveyor belt, which is electrically-earthed. The/each gun was fitted with a 2.5 mm flat spray nozzle. The powder coating booth was provided with a fluidizing hopper to
30 contain powder (the hopper being fitted with a venturi pump to supply the powder to the gun); a recovery drum to collect waste powder at the bottom of the booth; and an air

control unit for regulating the supply of fluidizing air to the hopper, and of flow and atomizing air to the pump and gun(s). The hopper, pump and recovery drum are all available from Nordson. The powder booth incorporated features that enabled the safe handling of fine powders (including air extraction through cartridge and HEPA filters, and a fire detection system).

Infra-red oven: a "Curemaster Super" oven with three 1 kW short-wave infra-red heaters, available from Trisk of Sunderland, Tyne and Wear, UK

Through-air ovens: a gas oven (4 meters long) and an electric oven (2 meters long), both available from Cavitec of Munchwilten, Switzerland.

Abrasive spray equipment: a spray booth equipped with one reciprocating spray gun, available from Charvot of Grenoble, France; and a spray booth equipped with two spray guns, available as Model 21 from Binks Manufacturing Company, of Illinois, USA.

15 Test methods

Schiefer cut test: This test provided a measure of the cut (material removed from a work piece) by an abrasive article under wet conditions. A 10.16 cm diameter circular specimen was cut from the abrasive material to be tested and secured by a pressure-sensitive adhesive to a back-up pad that had been pre-conditioned by soaking in water.

20 The abrasive material was also pre-wetted. The back-up pad was secured to the driven plate of a Schiefer Abrasion Tester (available from Frazier Precision Company.

Gaithersburg, Md.), which had been plumbed for wet testing. Circular acrylic plastic work pieces 10.16 cm diameter by 1.27 cm thick, available as "POLYCAST" acrylic plastic from Seelye Plastics, Bloomington, Min.. were employed. The initial weight of

25 each work piece was recorded to the nearest milligram prior to mounting on the work piece holder of the abrasion tester. The water drip rate was set to 60 ± 6 drops per

minute. A 4.55 Kg load was placed on the abrasion tester weight platform and the mounted abrasive specimen was lowered onto the work piece. The machine was set to run for 5000 cycles and then automatically stopped. After each 5000 cycles of the test,

30 the work piece was wiped free of water and debris and weighed. The cumulative cut for each 5000-cycle test was the difference between the initial weight and the weight following each test. **Tear test:** This test provided a measure of the tearing strength of

an abrasive article and was carried out on an ELMENDORF machine in the manner described in US Patents Nos. 1 423 841 and 1 423 842.

Load at break: This test provided a measure of the force needed (in the machine direction) to break a 51x135 mm sample of abrasive material and was carried out using
5 a "Type 1101 Dynamometer", available from Instron Corp. of Massachusetts, USA.

Example 1

A 30cm wide air-laid nonwoven web weighing 130 g/m² and comprising 20dtex nylon staple fibers was formed on the "Rando Webber" machine at a rate of 2 m/min. The
10 fibre bales had previously been pre-opened using the Laroche fibre opener. The web was conveyed in line on the horizontal metallic open mesh conveyor belt through the powder coating booth, where epoxy resin powder was directed at the web by a single "Versaspray II" spray gun located 30cm above the web. The powder was supplied to the spray gun from the hopper in which it was fluidized until gentle bubbling using air
15 at a pressure of 0.5 bar. The air pressure settings of the spray gun were 2-3 bar for the flow (or primary air) and 1-1.5 bar for the atomizing (or secondary) air and the maximum voltage (100 kV) was applied. Resin powder was deposited in the web at a weight of about 58g/m², and any epoxy resin powder that passed through the web was collected in the recovery drum, positioned underneath the open mesh conveyor belt.
20 The powdered web was then heated in the gas oven at 170°C for 2 min to fuse and cure the resin, using a low speed setting for the recirculating air to avoid dislodging the resin powder.

Abrasive particles were then applied to the bonded web in the following manner. An
25 abrasive-resin slurry was prepared by mixing together thoroughly corundum particles (55%), phenolic resin (20%), water (20%), filler (4%) and pigment (1%). The slurry was then transferred to the supply tanks of the spray booth equipped with one reciprocating spray gun. The bonded web was passed through the spray booth at a speed of 2m/min, and one side of the web was sprayed with the slurry at a weight in the
30 range of about 180 - 220 g/m². The web was then passed through the gas oven in which it was heated at 180°C for about 2 minutes to cure the phenolic resin. The web was then turned over and conveyed again through the spray booth and the gas oven with the

other surface of the web uppermost. The resulting nonwoven abrasive web was cut into abrasive hand-pads having a dimension of about 150 x 225 mm.

Example 2

- 5 Example 1 was repeated with the exception that the air-laid web was formed from a mixture of 20dtex and 80dtex nylon staple fibers (50% of each), and the amount of resin powder deposited on the web was 47 g/m².

Example 3

- 10 Example 2 was repeated with the epoxy resin powder replaced by copolyamide resin powder. The flow and homogeneity of the resin powder was improved by adding 0.5% of powder flow aid and by vibrating the hopper using a vibratory table. Fluidizing air was supplied to the hopper at a pressure of 1 bar, and the air pressure settings for the gun were 3 bar for the flow air and 4 bar for the atomizing air. The amount of resin
15 powder deposited on the web was 28 g/m².

Example 4

- A 30cm wide air-laid nonwoven web weighing 190 g/m² was formed from the sisal fibres on the "Rando Webber" machine at a rate of 2 m/min. The fibre bales had
20 previously been pre-opened using the Laroche fibre opener. The web was conveyed in line through the powder coating booth on the open mesh conveyor belt, where copolyamide resin powder (blended with 0.5% by weight of flow aid) was directed at the web by two "Versaspray II" spray guns, arranged one behind the other, that were fixed 30cm above the web and inclined on opposite sides of the vertical at an angle in
25 the range of 20° - 30°. The resin powder was supplied to the guns from the hopper, in which it was fluidized until gently bubbling using air at a pressure of 1.5 bar. The air pressure settings for the guns were 2 bar for the flow air and 1 bar for the atomizing air, and the maximum voltage (100 kV) was applied. Resin powder was deposited in the web at a weight of about 60 g/m² and any resin powder that passed through the web
30 was collected in the recovery drum, positioned underneath the open mesh conveyor belt. The powdered web was then heated in line, first in the infra-red oven at a temperature in the range of 150 – 160°C with the heaters positioned 3 cm above the

web to pre-set the resin powder and then in the electric oven at a temperature of 160°C using a low-speed setting for the recirculating air. The total residence time in the oven was 1 min.

- 5 The web was then turned over and conveyed again through the powder coating booth and the ovens with the other surface of the web uppermost.

Poly(vinylchloride) particles were then applied to the bonded web in the following manner. An abrasive-resin slurry was prepared by thoroughly mixing together the particles (25%) and the latex resin (68.5%) with the cross-linking agents (1.2% of (i) and 5.3% of (ii)). The slurry was then transferred to the supply tank of the spray booth having a single spray gun. The bonded web was passed through the spray booth at a speed of 2 m/min, and sprayed on one side with the slurry from the gun which was reciprocated across the web to ensure even coverage of the web with the slurry at a coating weight of about 300 g/m². The web was then passed through the gas oven in which it was heated at 180°C for 2 min. to cure the latex resin. The web was then turned over and conveyed again through the spray booth so that it was sprayed with slurry on the other side in the same manner. It was then again passed through the gas oven.

20

The resulting nonwoven abrasive web contained 150 g/m² of the poly(vinyl chloride) particles and was cut into domestic scouring pads having dimensions of about 75 x 90 mm.

25 **Example 5**

Example 4 was repeated, with the following modifications:

The nonwoven web weighed 150 – 170 g/m² and was formed from the coco fibres on the “Rando Webber” machine at a slower rate (1 m/min) to enable the curing time for the resin powder to be increased (see later). Before entering the powder coater, the web was humidified to increase its conductivity and, thereby, its uptake of resin powder.

30

The web was humidified using the water spray head which was supplied with water at a pressure of 1 bar and atomizing air at a pressure of 2.5 bar. The powder coater used a

single "Versaspray II" spray gun to direct epoxy resin powder at the web from a distance of 30 cm. The resin powder was fluidized in the hopper of the powder coater using air at a pressure of 1.8 bar. The air pressure settings for the guns were 1 bar for the flow air and 0.8 bar for the atomizing air. Resin powder was deposited in the web at a weight of 250 g/m². The infra-red heater was omitted and the powdered web was heated in the electric oven only, at a temperature of 170°C for 2 min., using a low speed setting for the recirculating air.

Corundum particles were then applied to the bonded web in the following manner. An abrasive-resin slurry was prepared by mixing together thoroughly the particles (25%) and the phenolic resin (75%). The slurry was then transferred to the supply tank of the spray booth having four spray guns. The bonded web was passed through the spray booth at a speed of 2 m/min, and sprayed on one side with the slurry from the guns to provide even coverage of the web with the slurry at a coating weight of about 230 - 260 g/m². The web was then passed through the gas oven in which it was heated at 180°C for 2 min. to cure the phenolic resin. The web was then turned over and conveyed again through the spray booth so that it was sprayed with slurry on the other side in the same manner. It was then again passed through the gas oven to yield a nonwoven scouring web which was cut into domestic scouring pads.

20

Results (Examples 1 to 3)

Samples of the abrasive hand-pads resulting from Examples 1 to 3 (Samples 1 to 3 respectively) were tested for abrasive performance. The results are shown in the following Table together with the results for a reference sample (REF) prepared as described in Example 1 except that the pre-bonded web was prepared using a liquid phenolic resin applied by roll coating, rather than epoxy resin powder applied as described.

25

Sample	Weight of sample (g/m ²)	Thickness of sample (mm)	Schiefer cut test for both sides of sample (gms)	Tear test	Load at break (N)
1	491	12.8	2.6/3.0	87	95.8
2	493	12.5	3.0/3.2	84	94.3
3	528	13.2	3.2/3.2	91	115.8

REF	560	10.5	2.8/3.0	45	90
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The Table above shows that the Samples 1 to 3 had good physical/functional properties and compared well with the reference sample. The tear strength of Samples 1 to 3 was particularly good, being about double that of the reference sample. The lower cut test results for Sample 1, compared with those for Samples 2 and 3, were considered to be due to the higher flexibility of this sample resulting from the fibres employed.

The Table also indicates that Samples 1 to 3 had a lower density (i.e. they were more open) than the reference sample because they were about 20% thicker although the initial fiber weights were about the same. This in turn indicates the possibility of producing effective abrasive products using a reduced amount of fibre. Moreover, the more open structure of Samples 1 to 3 means that they will be less likely to become clogged during use. The open structure is considered to be a consequence of the fact that the webs from which the Samples 1 to 3 were made were pre-bonded in a way that did not involve the web being subjected to pressure (e.g. as a result of contact by rollers).

Results (Examples 4 and 5)

Samples of the domestic scouring pads resulting from Examples 4 and 5 were used for cleaning soiled dishes in a simulated domestic environment and, based on a visual assessment, were found to perform at least as well as conventional synthetic scouring pads and, generally, better than traditional scouring pads made from natural fibres.

The scouring pads produced by the processes of Examples 4 and 5 offer the advantage that they can more easily be recycled after use since they are formed using natural vegetable fibres. Despite that, the homogeneity of the scouring pads is high compared with traditional natural fibre scourers making it possible to offer, to the consumer, an environmentally-friendly but comparatively standardized product. In addition, the scouring pads exhibit the advantageous openness of both traditional natural fibre scourers and conventional synthetic scourers, together with the abrasive performance of the latter. These advantages are considered to be a consequence of the fact that the

scouring pads comprise a mechanically-formed (dry-laid) web of natural fibres which is pre-bonded in a way that does not involve the web being subjected to pressure (e.g. as a result of contact by rollers) that could irreversibly compress or damage the web fibres.

- 5 An advantage of the processes described in all of the above Examples is that no volatile organic compounds (VOCs) are produced in the formation of the pre-bonded webs to which the resin/abrasive slurries are subsequently applied. In addition, the energy required in these processes to produce the pre-bonded webs may be less than that required if a liquid pre-bond resin were used. Consequently, the environmental effects
10 of the processes can be substantially less than those conventionally used to produce synthetic scouring materials.

It will be appreciated that, although the above Examples describe the production of abrasive hand pads and domestic scouring pads, methods in accordance with the
15 present invention could, with appropriate changes where necessary in the materials and process steps employed, be used to produce other abrasive articles including, for example, abrasive discs and floor pads for use in abrading machines.

CLAIMS

1. A method of making an abrasive article, including the steps of
(i) forming a three-dimensional nonwoven fibrous web contacted with
5 dry particulate material that includes fusible binder particles;
(ii) exposing the web to conditions that cause the binder particles to form a flowable
liquid binder, and then solidifying the liquid binder to form bonds between the fibres of
the web and thereby provide a pre-bonded web; and
(iii) applying abrasive particles to the pre-bonded web, and bonding the abrasive
10 particles to the fibres of the pre-bonded web to provide the abrasive article.
2. A method as claimed in claim 1, in which step (i) comprises forming fibres into a
three-dimensional nonwoven web, and then contacting the web with the dry particulate
15 material.
3. A method as claimed in claim 1, in which step (i) comprises mixing fibres with the
dry particulate material, and then forming the mixture into a three-dimensional
nonwoven web.
- 20 4. A method as claimed in any one of the preceding claims, in which the fibres of the
web comprise synthetic fibres or natural fibres or mixtures thereof.
5. A method as claimed in claim 4, in which the fibres of the web comprise
polyamide fibres.
25
6. A method as claimed in claim 4, in which the fibres of the web comprises coco, sisal
and/or hemp fibres.
7. A method as claimed in any one of the preceding claims, in which the fusible binder
30 particles comprise thermosetting or thermoplastic materials.

8. A method as claimed in claim 7, in which the binder particles are selected from the group consisting of: epoxy resins, copolyamides, and copolyesters.
9. A method as claimed in any one of the preceding claims, in which the binder
5 particles have a particle size of less than 200µm.
10. A method as claimed in any one of the preceding claims, in which the binder particles are applied to the web without applying a compressive force to the web.
- 10 11. A method as claimed in any one of the preceding claims, in which the binder particles are deposited across the whole thickness of the web under the action of an electrostatic force.
12. A method as claimed in any one of the preceding claims, in which an electrostatic
15 charge is applied to the binder particles, which are then directed towards the web while the latter is located on an electrically-grounded support surface.
13. A method as claimed in any one of the preceding claims, in which the web is exposed to heat and/or moisture to cause the binder particles to form the flowable
20 liquid binder.
14. A method as claimed in any one of the preceding claims, in which the abrasive particles are bonded to the fibres of the pre-bonded web by at least a make-coat binder resin.
25
15. A method as claimed in claim 14, in which the make-coat binder resin is applied to the pre-bonded web in liquid form.
16. A method as claimed in claim 14 or claim 15, in which the abrasive particles are
30 applied to the pre-bonded web at the same time as the make-coat binder resin.
17. A method as claimed in claim 15, in which the abrasive particles and the make-coat

binder resin are applied to the pre-bonded web together as a slurry.

18. A method as claimed in claim 17, in which the slurry is sprayed onto the pre-bonded web.

5

19. A method as claimed in any one of claims 14 to 18, in which the make-coat binder resin is selected from the group consisting of: latex resins, and phenolic resins.

20. A method as claimed in any one of the preceding claims, in which the pre-bonded web has a maximum density of 50 kg/m^3 .

10

21. A method as claimed in claim 20, in which the pre-bonded web has a maximum density of 30 kg/m^3 .

22. A method as claimed in any one of the preceding claims, in which the pre-bonded web has a minimum thickness of 5 mm.

15

23. A method as claimed in any one of the preceding claims, in which the nonwoven fibrous web is formed by a dry-laying process.

20

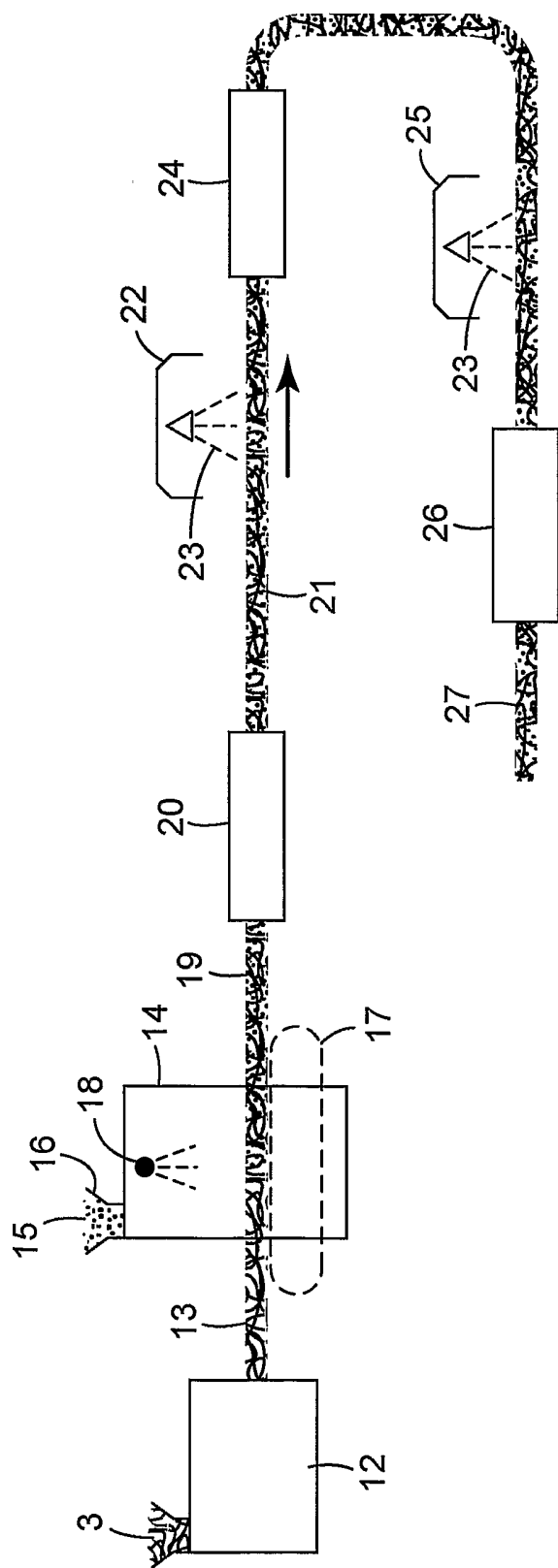


FIG. 1

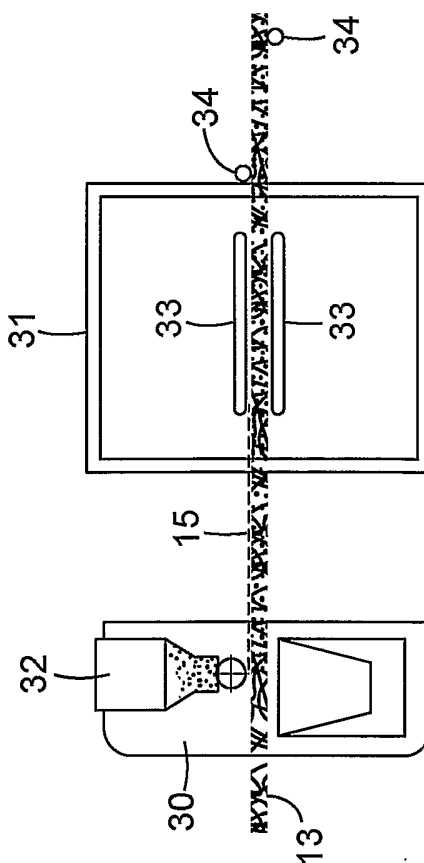


FIG. 2

INTERNATIONAL SEARCH REPORT

International Application No
PCT/US2004/010840

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 B24D11/00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 B24D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 5 573 619 A (BENEDICT HAROLD W ET AL) 12 November 1996 (1996-11-12) column 47, line 20 - line 41 column 10, line 4 - line 18 column 19, line 49 - line 64 column 22, line 25 - line 38	1-23
Y	US 5 565 049 A (KING DOUGLAS D ET AL) 15 October 1996 (1996-10-15) column 1, line 12 - line 17; claims 1-7; figure 1 column 4 - column 6	1-23

Further documents are listed in the continuation of box C.

Patent family members are listed in annex.

° Special categories of cited documents :

- *A* document defining the general state of the art which is not considered to be of particular relevance
- *E* earlier document but published on or after the international filing date
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- *X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
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Date of the actual completion of the international search

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INTERNATIONAL SEARCH REPORT

International Application No
PCT/US2004/010840

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 5 681 361 A (SANDERS JR RUFUS C) 28 October 1997 (1997-10-28) cited in the application column 4, line 15 - column 5, line 9 column 6, line 3 - line 68 column 8, line 65 - column 9, line 15 -----	1-4,6-9
A	US 5 626 512 A (PALAIKIS LIANA V ET AL) 6 May 1997 (1997-05-06) column 9, line 50 - column 10, line 19 -----	1,11,12
A	US 3 286 007 A (DE ROCHE LEO JOHN ET AL) 15 November 1966 (1966-11-15) claims 1-4; figure 1 -----	1-3,7,10
A	US 4 886 701 A (EHNERT GERD ET AL) 12 December 1989 (1989-12-12) column 4, line 55 - column 5, line 19 -----	3

INTERNATIONAL SEARCH REPORT

International Application No
PCT/US2004/010840

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
US 5573619	A	12-11-1996	US 5609706 A	11-03-1997
			US 6406576 B1	18-06-2002
			US 6406577 B1	18-06-2002
			AT 176883 T	15-03-1999
			AU 3058192 A	28-07-1993
			BR 9206806 A	31-10-1995
			CA 2116686 A1	08-07-1993
			CN 1073388 A ,B	23-06-1993
			DE 69228487 D1	01-04-1999
			DE 69228487 T2	02-09-1999
			EP 0619769 A1	19-10-1994
			JP 7502458 T	16-03-1995
			KR 161543 B1	15-12-1998
			MX 9206921 A1	01-06-1993
			NO 942334 A	20-06-1994
			RU 2116186 C1	27-07-1998
			WO 9312911 A1	08-07-1993
			US 6066188 A	23-05-2000
			ZA 9208835 A	06-08-1993
US 5565049	A	15-10-1996	CA 2102361 A1	24-01-1995
US 5681361	A	28-10-1997	DE 69607037 D1	13-04-2000
			DE 69607037 T2	19-10-2000
			EP 0874716 A1	04-11-1998
			JP 2000502960 T	14-03-2000
			WO 9725185 A1	17-07-1997
US 5626512	A	06-05-1997	AU 5317196 A	21-11-1996
			BR 9608205 A	01-06-1999
			CA 2218956 A1	07-11-1996
			CN 1183817 A	03-06-1998
			EP 0823947 A1	18-02-1998
			JP 2000508926 T	18-07-2000
			WO 9635011 A1	07-11-1996
			ZA 9603106 A	20-10-1997
US 3286007	A	15-11-1966	BE 669713 A	16-03-1966
			FR 1464666 A	06-01-1967
			GB 1124482 A	21-08-1968
			NL 6512240 A	22-03-1966
US 4886701	A	12-12-1989	DE 3704035 A1	03-03-1988
			AR 241164 A1	30-12-1991
			AT 69072 T	15-11-1991
			AU 600514 B2	16-08-1990
			AU 7770987 A	03-03-1988
			BR 8704484 A	26-04-1988
			CA 1330637 C	12-07-1994
			DE 3774214 D1	05-12-1991
			EP 0258684 A2	09-03-1988
			ES 2027666 T3	16-06-1992
			IN 168986 A1	03-08-1991
			JP 2097362 C	02-10-1996
			JP 8014066 B	14-02-1996
			JP 63135550 A	07-06-1988
			MX 159961 A	17-10-1989
			PT 85623 A ,B	14-10-1988

INTERNATIONAL SEARCH REPORT

International Application No
PCT/US2004/010840

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 4886701 A		TR 28650 A ZA 8706489 A	27-12-1996 29-02-1988
<hr/>			