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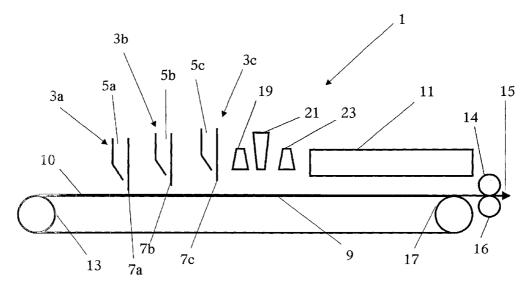
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(54) Title: FLOOR COVERING



WO 03/100162 A2 (57) Abstract: The invention relates to a plastics floor covering with enhanced slip resistance having improved stain resistance without affecting the effectiveness of the life of non-slip properties. The floor covering is non-laminated and includes a first layer of plastics material having an upper surface and a first particulate material embedded in the layer of plastics material and exposed at the upper surface to provide slip resistance, wherein the layer of plastics material is substantially free from a plasticiser.



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## FLOOR COVERING

The present invention provides a new slip resistant floor covering which is stain resistant and a method for manufacturing the floor covering.

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To provide enhanced slip resistance, a floor covering is generally manufactured having a roughened surface which can be created by embossing its surface and/or by the addition of particulate material to the main component of the floor covering during manufacture.

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The roughened surface of the flooring increases the risk of dirt being trapped on the surface. The dirt, depending on its nature, can be compatible with components of the flooring resulting in absorption of the dirt into the surface giving rise to a stain.

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In WO 00/42274 and WO 00/22225 this problem was overcome by applying a coating portion to the base layer of the floor covering to impart improved stain resistance. However it has been found that the efficiency of the coating portion at resisting staining decreases if it is contaminated by chemicals from the base portion which can occur during its manufacture or subsequently due to migration.

A way of ameliorating these problems has been sought.

According to a first aspect of the invention there is provided a non-laminated plastics floor covering including a first layer of plastics material having an upper surface and a first particulate material embedded in the layer of plastics material and exposed at the upper surface to provide slip resistance, wherein the layer of plastics material is substantially free from a plasticiser.

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The invention therefore provides a floor covering with enhanced slip resistance having improved stain resistance without affecting the effectiveness of the life of non-slip properties which are achieved by including the particulate material.

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The floor covering according to the invention is non-laminated which generally means that it is not formed from separate layers of plastics material which have been laminated together. Problems with floor coverings manufactured in this way include de-lamination of the layers and retention of the particulate material. It has been found that when a floor covering is formed from laminated layers there is a problem with sufficiently embedding the particulate material in the layers so that the floor covering is slip resistant.

According to a second aspect of the invention there is provided a method of making a slip-resistant floor covering, the method including the steps of:-

applying a layer of a plastics material which is substantially free from a plasticiser;

applying a first particulate material; melting or curing the plastics material.

According to the invention there is also provided a slip-resistant floor covering obtainable by the method of the present invention.

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The method according to the second aspect of the invention is preferably a method of making a floor covering according to the invention.

The layer of plastics material may be applied to a belt from which the 30 floor covering made by the method can be removed, e.g. an optionally glass filled PTFE belt support. Alternatively, where the floor covering

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includes a support, the first layer may be applied to a support. The support generally has an upper surface and a lower surface. Where the first layer is applied to the support, it is applied to the upper surface. The lower surface of the support is optionally coated with a second layer of plastics material before or after the method of the invention is performed.

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Preferably the floor covering according to the invention consists essentially of a support having an upper surface and a lower surface wherein on the upper surface of the support there is a first layer of plastics material having an upper surface and a first particulate material embedded in the first layer of plastics material and exposed at the upper surface of the plastics material to provide slip resistance and on the lower surface of the support there is optionally a second layer of plastics material.

Optionally the method also includes the step of applying the layer of plastics material which is substantially free from a plasticiser in two or more stages such that the composition and/or properties of the plastics material in the layer can be varied. The layer of plastics material may therefore have a lower part applied in a first stage and upper part(s) applied in a second or subsequent stage. For example the plastics material used to form the upper part may optionally have better wear resistance, greater scratch resistance and/or improved surface hardness than the plastics material applied in the first stage.

Where the layer of plastics material is applied in two or more stages, the method of the invention preferably includes one or more steps of cohering the layer of plastics material after application of one stage and before a further stage is applied to reduce scattering of the plastics material and mixing between the different parts. The cohering step generally involves

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partially sintering the particles of the plastics material (e.g. with heat and/or radiation). This is in order to prevent the powder being blown off the layer of plastics material on entering the oven. Where the plastics material is a thermoplastic material, the cohering step preferably involves the application of heat and/or infrared radiation. The plastics material is optionally cooled after the cohering step and before the application of a further stage.

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Optionally the plastics material applied in the method of the invention includes an ionomer and/or a foaming agent. Preferably where two or more stages are used to apply the plastics material, the plastics material applied in the second or subsequent stage includes an ionomer. The plastics material applied in the first stage preferably includes a foaming agent such that the lower part of the layer of plastics material is flexible.

The first particulate material preferably penetrates the lower part of the layer which is useful as it means that the particulate material is more securely embedded in the floor covering.

An ionomer is a copolymer of an olefin monomer and a polymerisable carboxylic acid monomer (e.g. acrylic acid or methacrylic acid) which has been at least partially neutralised by a metal cation such as sodium, lithium, zinc or magnesium. The bivalent metal ions reversibly cross-link adjacent carboxylate groups and therefore modify the properties of the polymer or copolymer. Different methods for the preparation of ionomers are described in European patent application EP-A-0 847 406.

According to a third aspect of the invention there is provided a method of making a floor covering including a layer of plastics material wherein a portion of the plastics material includes an ionomer which method includes the steps of

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applying at least a part of a layer of plastics material including a copolymer of an olefin monomer and a polymerisable carboxylic acid monomer;

applying a compound including a metal cation to the surface of the part of the layer of plastics material;

curing the plastics material such that the metal cation at least partially neutralises the carboxylic acid monomer.

The advantage of the method according to the third aspect of the invention is that the portion of the plastics material which includes an ionomer can be carefully controlled. Including an ionomer in a floor covering is advantageous because it increases the wear resistance. The particulate material included in the floor covering according to the invention provides wear resistance but for some applications, for example where the floor covering may be subjected to heavy traffic, it may be desirable to improve the wear resistance by including an ionomer. However inclusion of an ionomer also increases the stiffness of a floor covering. This is a potential problem because it is important for a floor covering to be flexible especially as it is normally supplied in a roll. By effectively limiting the formation of the ionomer to the surface of the part of the layer of plastics material, the advantage of increased wear resistance at that surface is obtained without the disadvantage of increased stiffness throughout that part of the layer which would normally occur if the plastics material applied included a pre-formed ionomer.

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Preferably the portion of the layer of plastics material which includes an ionomer is an upper portion of the layer, more preferably it is the upper surface of the layer. Preferably the said compound is applied by sprinkling, spraying or spreading.

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The method of the third aspect of the invention may optionally be included as steps of the method according to the second aspect of the invention.

The compound including a metal cation is preferably a salt of a metal cation or, more preferably, a base or metal oxide. Bases which are suitable for use as the said compound are those which are strong enough to neutralise the carboxylic acid groups of the monomer. Examples of bases include NaOH, KOH, Mg(OH)<sub>2</sub>, Ca(OH)<sub>2</sub> or Zn(OH)<sub>2</sub>. Examples of metal oxides include MgO, CaO, ZnO, K<sub>2</sub>O. Suitable salts include silicates such as Na<sub>2</sub>SiO<sub>3</sub>, carbonates such as Na<sub>2</sub>CO<sub>3</sub>, CaCO<sub>3</sub>, borates (such as sodium borate, potassium borate etc), and/or phosphates.

A suitable foaming agent is an azodicarbonamide or a gas filled 15 microsphere such as that marketed under the name Expancel by Akzo Nobel.

The plastics material preferably further includes additives commonly used in the art such as a UV stabiliser system, a biocide, a dry flow aid such as fumed silica, and/or a static dissipator.

The plastics material used in the invention is preferably substantially free from PVC. More preferably, the plastics material used in the invention is a thermoplastic polymer and/or a thermoplastic elastomer. More preferably it is a thermoplastic polymer, particularly a thermoplastic polymer such as EVA (an ethylene vinyl acetate copolymer), a polyethylene-acrylic acid copolymer, a terpolymer (e.g. of ethylene/alkyl acrylate/maleic anhydride), and/or a copolymer of ethylene and methacrylic acid (e.g. that sold under the brand name Nucrel by Du Pont).

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Examples of suitable thermoplastic elastomers include: styrene triblock copolymers (e.g. polystyrene polybutadiene polystyrene block copolymers or polystyrene polyisoprene polystyrene block copolymers), blends of thermoplastic elastomers and thermoplastic polymers (e.g. mixtures of ethylene-propylene rubber and crystalline polypropylene, blends of polypropylene and EPDM (ethylene propylene diene monomer) or blends of polypropylene and natural rubber (with in-situ peroxide-initiated crosslinking)), thermoplastic polyurethanes (polyester or polyether based), thermoplastic copolyesters, and/or polyether-polyamide block copolymers.

Where the layer of plastics material is applied in two or more stages, the plastics material applied in the first stage preferably includes a second particulate material dispersed therein to further improve the non-slip properties of the flooring covering and/or to enhance the hardness and/or the wear resistance of the flooring covering during the life of the product.

The floor covering may optionally contain decorative elements such as a pigment, pigmented particles (e.g. pigmented silicon carbide particles), optionally coloured or transparent silica chips and/or fine particles, and/or optionally coloured plastics chips. Preferably the plastics chips are formed from the plastics material used to form the floor covering.

The floor covering preferably includes a support, especially a reinforcing support; the support is preferably a glass fibre reinforced non-woven support. The support generally has an upper and a lower surface. The first layer of plastics material is applied to the upper surface of the support; a second layer of plastics material is optionally applied to the lower surface of the support.

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The first and/or second particulate material is preferably a grit; more preferably it is one or more of a number of types of hard particles including silicon carbide, a silica (e.g. quartz, a coloured or natural sand or a flint), aluminium oxide and/or emery.

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The melting or curing step will normally involve the application of heat e.g. by forced air convection optionally with the application of infra red radiation.

10 Before the first particulate material is applied, the method preferably includes a step of cohering the plastics material. The plastics material is preferably cohered by the application of infra red heat. This step is advantageous as it alleviates the problem of 'craters' being formed on the upper surface of the plastics material when the first particulate material is applied. As an alternative, the first particulate material is optionally applied in a manner such that its kinetic energy on impact with the plastics material is minimised, e.g. by applying the first particulate material through a sieve arranged close to the upper surface of the plastics material.

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If the plastics material has not been sintered prior to application of the surface grits, then it should preferably be sintered before it is melted or cured. The plastics material is preferably sintered by the application of infra red heat. This step is particularly useful when a convection oven is used in the curing step. This is because there can be strong air-flows in a convection oven which can potentially blow the plastics material around the oven, particularly when the plastics material is applied in powder form.

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After curing, the method optionally includes the steps of embossing the floor covering, cooling the floor covering and/or winding it up and trimming it to length.

- The plastics material is preferably applied in the form of a powder, granules or pellets; more preferably in the form of a powder. Preferably it is applied to the desired thickness using knife over roller or knife over air.
- 10 The plastics material is optionally a thermoplastic plastics material, for example a thermoplastic elastomer.

Preferably the floor covering material is embossed.

The present invention will now be described by means of example only with reference to the drawings in which:

Figure 1 shows a production line suitable for use in the method of manufacturing according to the invention; and

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Figure 2 shows a schematic cross-sectional view of a floor covering according to the invention.

Figure 1 shows a schematic cross-sectional view of key elements of a production line 1. The line 1 has a belt 9 which runs from roller 13 past roller 17 in direction 15. The belt 9 is in the form of a glass re-inforced closed mesh PTFE filled material. A scrim 10 is provided on belt 9. The scrim 10 is in the form of a non-woven glass fibre support for the floor covering. The scrim 10 is optionally coated with plastics material, particularly on its reverse side. As an alternative the scrim 10 may be

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provided in the form of a glass fibre crenette over a cellulose/polyester non-woven support.

There are three dispensers 3a,3b,3c arranged sequentially near the start of production line 1. Each dispenser 3a,3b,3c has a receptacle for plastics material 5a,5b,5c and a doctor blade 7a,7b,7c. The production line 1 also includes an infra-red heater 19, a scatter head for grits 21, a second infra-red heater 23, a curing oven 11 and a patterned emboss roller 14 over a smooth rubber nip roller 16.

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In use, in a first procedure for operating production line 1 where a floor covering having two layers is to be made, the receptacles 5a,5b of the first two dispensers 3a,3b are filled with plastics material. Dispenser 3a supplies a layer of the first plastics material onto the belt which is levelled to a first height by doctor blade 7a. Dispenser 3b supplies a layer of a second plastics material onto the first plastics material. The layer of a second plastics material is levelled to a second height by doctor blade 7b.

The two layers of plastics material are then cohered by the application of infra-red radiation to the upper surface of the second plastics material by infra-red heater 19. A particulate material in the form of grits is then applied to the cohered upper surface of the second plastics material by scatter head 21. The combination of layers of plastics material and particulate material is then sintered by the application of further infra-red radiation by infra-red heater 21 before being cured in the oven 11 to form a floor covering which is then nipped between an embossing roller 14 and a rubber roller 16 before being cut to length and rolled up.

30 In use, in a second procedure for operating production line 1 where a floor covering having two layers is to be made, the receptacles 5a,5b of

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the first two dispensers 3a,3b are filled with plastics material. Dispenser 3a supplies a layer of a first plastics material onto the belt which is levelled to a first height by doctor blade 7a. The layer of plastics material is then cohered by the application of infra-red radiation to its upper surface by infra-red heater 19. The coated scrim thus formed is cooled and then put through the production line a second time.

Dispenser 3b supplies a layer of a second plastics material onto the first layer of plastics material. The layer of the second plastics material is levelled to a second height by doctor blade 7b.

The second layer of plastics material is then cohered by the application of infra-red radiation to the upper surface of the second plastics material by infra-red heater 19. A particulate material in the form of grits is then applied to the cohered upper surface of the second plastics material by scatter head 21. The combination of layers of plastics material and particulate material is then sintered by the application of further infra-red radiation by infra-red heater 21 before being cured in the oven 11 to form a floor covering which is then nipped between an embossing roller 14 and a rubber roller 16 before being cut to length and rolled up.

A schematic cross-section of a floor covering 31 according to the invention is shown in Figure 2. Floor covering 31 includes a base portion 35, an upper portion 37 and a support 53. Particulate material 41 in the upper portion 37 penetrates the base portion 35 and is proud of the upper surface of the upper portion 37. Particulate material 43 is also in the upper portion 37 and is substantially smaller than particulate material 41. Particulate material 43 is also proud of the upper surface of the upper portion 37.

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Examples of plastics materials suitable for use in the invention follow.

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# PREPARATION EXAMPLE 1

The powder formulations detailed in Table 1 were prepared for use in Examples 2 to 5.

TABLE 1

	Powder Formulations			
	A	В	С	
	Weight/kg	Weight/kg	Weight/kg	
Nucrel 925	••	100	25	
Nucrel 960	100	-	-	
Surlyn 9910		-	75	
ON320	25	30	20	
Irganox 1010	0.1	0.1	0.1	
Tinuvin 770	0.2	0.2	0.2	
Tinuvin 328	0.2	0.2	0.2	
Blue BLP	0.4	0.2	0.2	
Expancel 098 DU120	-	0.5	-	

wherein Nucrel 925 and 960 are copolymers of ethylene and methacrylic acid supplied by DuPont. Surlyn 9910 is an ionomer also supplied by DuPont. ON320 is a grade of aluminium trihydrate supplied by OMYA. Irganox 1010, Tinuvin 770 and Tinuvin 328 are antioxidants supplied by Ciba Geigy. Expancel DU120 are microspheres (containing isopentane) supplied by Akzo Nobel.

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## **EXAMPLE 2**

The ingredients of formulation A were weighed and blended by being tumbled together. The blend was extruded in a Werner and Pfleiderer extruder (model number ZSK 70) with the feed zone temperature set at  $160^{\circ}$ C, and the barrel/die set at  $200^{\circ}$ C. The extrudate was water cooled and pelletized. The pellets were dried in a vacuum drier at  $40^{\circ}$ C and then cryogenically ground in an Alpine pin disc mill using a single pass and no intermediate sieving screen. The temperature of the material prior to its introduction in to the mill was  $-150^{\circ}$ C. The mill was maintained at  $-150^{\circ}$ C during grinding. 99% of the resulting powder was of a size below  $250~\mu m$ . The resultant powder was blended by tumble mixing with 0.2% aluminium oxide C supplied by Degussa corporation.

15 The resultant powder was then spread coated on to a scrim to a thickness of 5 mm by knife over roller. The scrim was a 2 m width cellulose/polyester support (Dexter 555:030) reinforced with a Kirson '3x3' 68 tex glass crennette moving at a rate of 5 m/minute. Particles of coloured quartz of a size in the range 0.7 - 1.2 mm were then scattered 20 on to the surface of the powder at a rate of 100 g/m<sup>2</sup>. The coated scrim was then passed under a 50 kW medium wave infra red heater (height 10 cm above the coated scrim; width 2.5 m; length 1 m). The power output was adjusted in order to partially sinter the powder. Particles of silicon carbide size F36 (FEPA Standard 42-GB-1984) were then scattered 25 on to the surface at a rate of 100 gm<sup>-2</sup>. The coated scrim was then passed in to a convection oven where it was exposed to a temperature of 190°C for 2.5 minutes before being embossed, cooled and wound up for subsequent trimming to size.

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# EXAMPLE 3

The ingredients of formulation B were weighed and blended by being tumbled together. The blend was extruded in a Werner and Pfleiderer extruder (model number ZSK 70) with the feed zone temperature set at  $160\,^{\circ}$ C, the barrel set at  $190\,^{\circ}$ C and the die at  $200\,^{\circ}$ C. The extrudate was water cooled and pelletized. The pellets were dried in a vacuum drier at  $40\,^{\circ}$ C and then cryogenically ground in an Alpine pin disc mill using a single pass and no intermediate sieving screen. The temperature of the material prior to its introduction in to the mill was -120 $\,^{\circ}$ C. The mill was maintained at -120 $\,^{\circ}$ C during grinding. 99% of the resulting powder was of a size below 400  $\mu$ m.

The resultant powder was then applied on to a scrim to a thickness of 2 mm by using knife over roller. The scrim was a 2 m width cellulose/polyester support (Dexter 555:030) reinforced with a Kirson '4x4' 34 tex glass crennette moving at a rate of 3 m/minute. The coated scrim was then passed under a 50 kW medium wave infra red heater (height 10 cm above the coated scrim; width 2.5 m; length 1 m). The power output was adjusted in order to partially sinter the powder. Particles of quartz of a size in the range 0.4-0.8 mm were then scattered on to the surface of the powder at a rate of 100 gm<sup>-2</sup>. The coated scrim was then passed in to a convection oven where it was exposed to a temperature of 195°C for 2.5 minutes before being embossed and cooled.

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The scrim was then further coated with powder of formulation A (made as described in Example 2). Formulation A was spread on to the resulting scrim to an additional thickness of 3 mm by knife over roller. This time the powder of formulation A had been pre-blended by tumbling with 10% (w/w) aluminium oxide (Washington Mills) size F40 (FEPA Standard 42-GB-1984) and 5% (w/w) Activox B (zinc oxide) supplied by Elementis

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Specialities. The coated scrim was then passed under a 50 kW medium wave infra red heater (height 10 cm above the coated scrim; width 2.5 m; length 1 m). The power output was adjusted in order to partially sinter the powder. Particles of coloured quartz of a size in the range 0.4-0.8 mm were then scattered on to the surface of the powder at a rate of 100 gm<sup>-2</sup>. Particles of silicon carbide size F40 (FEPA Standard 42-GB-1984) were then scattered on to the surface at a rate of 100 g m<sup>-2</sup>. The coated scrim was then passed in to a convection oven where it was exposed to a temperature of 200°C for 2.5 minutes before being embossed, cooled and wound up for subsequent trimming to size.

The product was then tested for slip resistance in wet conditions according to the UK Slip Resistance Guidelines. (The measurement of floor slip resistance: Guidelines recommended by the UK slip resistance group ISBN 1-85957-227-8) and shown to have a slip resistance value (SRV) in excess of 40 (coefficient of friction ca 0.4).

### **EXAMPLE 4**

The ingredients of formulation C were weighed and blended by being tumbled together. The blend was extruded in a Werner and Pfleiderer extruder (model number ZSK 70) with the feed zone temperature set at  $160^{\circ}$ C, and the barrel/die set at  $200^{\circ}$ C. The extrudate was water cooled and pelletized. The pellets were dried in a vacuum drier at  $40^{\circ}$ C and then cryogenically ground in an Alpine pin disc mill using a single pass and no intermediate sieving screen. The temperature of the material prior to its introduction in to the mill was -150°C. The mill was maintained at -150°C during grinding. 99% of the resulting powder was of a size below  $400~\mu m$ .

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The resultant powder was then spread coated on to a scrim to a thickness of 5 mm by knife over roller. The scrim was a 0.5 m width cellulose/polyester support (Dexter 555:030) reinforced with a Kirson '3x3' 34 tex glass crennette moving at a rate of 1 m/minute. Particles of coloured quartz of a size in the range 0.7-1.2 mm were then scattered on to the surface of the powder at a rate of 100 g/m<sup>2</sup>. The scrim was then passed under a 4 kW medium wave infra red heater (height 5 cm above the coated scrim; width 0.6m; length 0.4m). The power output was adjusted in order to partially sinter the powder. Particles of silicon carbide size F40 (FEPA Standard 42-GB-1984) were then scattered on to the surface at a rate of 100 gm<sup>-2</sup>. The coated scrim was then passed in to a convection oven where it was exposed to a temperature of 190°C for 3 minutes before being embossed, cooled and wound up for subsequent trimming to size.

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## **EXAMPLE 5**

The ingredients of formulation A were weighed and blended by being tumbled together. The blend was extruded in a Werner and Pfleiderer extruder (model number ZSK 70) with the feed zone temperature set at  $160^{\circ}$ C, and the barrel/die set at  $200^{\circ}$ C. The extrudate was water cooled and pelletized. The pellets were dried in a vacuum drier at  $40^{\circ}$ C and then cryogenically ground in an Alpine pin disc mill using a single pass and no intermediate sieving screen. The temperature of the material prior to its introduction in to the mill was  $-150^{\circ}$ C. The mill was maintained at  $-150^{\circ}$ C during grinding. 99% of the resulting powder was of a size below  $250~\mu m$ .

The resultant powder was then applied on to a scrim to a thickness of 5mm by using a Cavistat 91 Pulverstreuaggregat conventional powder scatter head (manufactured by Cavitec). The scrim was a 0.5 m width

cellulose/polyester support (Dexter 555:030) reinforced with a Kirson '4x4' 34 tex glass crennette moving at a rate of 1 m/minute. The scrim was then passed under a 4 kW medium wave infra red heater (height 5cm above the coated scrim; width 0.6m; length 0.4m). The power output was adjusted in order to partially sinter the powder. Particles of quartz of a size in the range 0.4-0.8mm were then scattered on to the surface of the powder at a rate of 100 g m<sup>-2</sup>. Particles of silicon carbide size F40 (FEPA Standard 42-GB-1984) were then scattered on to the surface at a rate of 100 gm<sup>-2</sup>. The coated scrim was then passed in to a convection oven where it was exposed to a temperature of 195°C for 3 minutes before being embossed and cooled.

The product was then tested for slip resistance in wet conditions according to the UK Slip Resistance Guidelines (The measurement of floor slip resistance: Guidelines recommended by the UK slip resistance group ISBN 1-85957-227-8) and shown to have a slip resistance value (SRV) in excess of 40 (coefficient of friction ca 0.4)

# PREPARATION EXAMPLE 6

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The powder formulations detailed in Table 2 were prepared for use in Examples 7 to 10. The amount of each ingredient is given in terms of its weight in kg.

TABLE 2

	Powder formulations			
	D	Е	F	G
Desmopan KU2-8651	100		,	
Arnitel EM400		100		

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Pebax 5533-SA-00			100	
Alcryn ALC3075 NC				100
ON320	20	20	20	20
Blue BLP	0.1	0.1	0.1	0.1

wherein Desmopan is a polyurethane supplied by Bayer. Arnitel is a polyetherester supplied by DSM. Pebax is a polyetheramide supplied by Atofina. Alcryn is an olefinic type TPE supplied by DuPont. ON320 is aluminium trihydrate supplied by OMYA. Blue BLP is a phthalocyanine blue pigment supplied by Ciba Pigments.

### **EXAMPLE 7**

The ingredients of formulation D were weighed and blended by being tumbled together. The blend was extruded in an extruder with a single screw with L:D ratio of 20 and a compression ratio of 1:3, maintaining the melt temperature in the region of 180-200°C. The extrudate was water cooled and pelletized. The pellets were dried in a vacuum drier at 80°C and then cryogenically ground in an Alpine pin disc mill using a single pass and no intermediate sieving screen. The temperature of the material prior to its introduction in to the mill was -120°C. The mill was maintained at -120°C during grinding. 99% of the resulting powder was of a size below 400 μm.

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The resultant powder was then applied on to a scrim to a thickness of 5mm by using a Cavistat 91 Pulverstreuaggregat conventional powder scatter head (manufactured by Cavitec). The scrim was a 0.5m width cellulose/polyester support (Dexter 555:030) reinforced with a Kirson '4x4' 34 tex glass crennette moving at a rate of 1 m/minute. The coated scrim was then passed under a 4 kW medium wave infra red heater

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(height 5cm above the coated scrim; width 0.6m; length 0.4m). The power output was adjusted in order to partially sinter the powder. Particles of quartz of a size in the range 0.4-0.8mm were then scattered on to the surface of the powder at a rate of 100 gm<sup>-2</sup>. Particles of silicon carbide size F40 (FEPA Standard 42-GB-1984) were then scattered on to the surface at a rate of 100 gm<sup>-2</sup>. The coated scrim was then passed in to a convection oven where it was exposed to a temperature of 200°C for 3 minutes before being embossed and cooled.

The product was then tested for slip resistance in wet conditions according to the UK Slip Resistance Guidelines (The measurement of floor slip resistance: Guidelines recommended by the UK slip resistance group ISBN 1-85957-227-8) and shown to have a slip resistance value (SRV) in excess of 40 (coefficient of friction ca 0.4)

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## **EXAMPLE 8**

The ingredients of formulation E were weighed and blended by being tumbled together. The blend was extruded in an extruder with a single screw with L:D ratio of 20 and a compression ratio of 1:3, maintaining the melt temperature in the region of 180-200°C. The extrudate was water cooled and pelletized. The pellets were dried in a vacuum drier at 60°C and then cryogenically ground in an Alpine pin disc mill using a single pass and no intermediate sieving screen. The temperature of the material prior to its introduction in to the mill was -140°C. The mill was maintained at -140°C during grinding. 99% of the resulting powder was of a size below 400  $\mu$ m.

The resultant powder was then applied on to a scrim to a thickness of 5mm by using a Cavistat 91 Pulverstreuaggregat conventional powder scatter head (manufactured by Cavitec). The scrim was a 0.5m width

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cellulose/polyester support (Dexter 555:030) reinforced with a Kirson '4x4' 34 tex glass crennette moving at a rate of 1 m/minute. The scrim was then passed under a 4 kW medium wave infra red heater (height 5cm above the coated scrim; width 0.6m; length 0.4m). The power output was adjusted in order to partially sinter the powder. Particles of quartz of a size in the range 0.4-0.8mm were then scattered on to the surface of the powder at a rate of 100gm<sup>-2</sup>. Particles of silicon carbide size F40 (FEPA Standard 42-GB-1984) were then scattered on to the surface at a rate of 100gm<sup>-2</sup>. The coated scrim was then passed in to a convection oven where it was exposed to a temperature of 200°C for 3 minutes before being embossed and cooled.

The product was then tested for slip resistance in wet conditions according to the UK Slip Resistance Guidelines (The measurement of floor slip resistance: Guidelines recommended by the UK slip resistance group ISBN 1-85957-227-8) and shown to have a slip resistance value (SRV) in excess of 40 (coefficient of friction ca 0.4).

### EXAMPLE 9

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The ingredients of formulation F were weighed and blended by being tumbled together. The blend was extruded in an extruder with a single screw with L:D ratio of 20 and a compression ratio of 1:3, maintaining the melt temperature in the region of 180-200°C. The extrudate was water cooled and pelletized. The pellets were dried in a vacuum drier at  $80^{\circ}$ C and then cryogenically ground in an Alpine pin disc mill using a single pass and no intermediate sieving screen. The temperature of the material prior to its introduction in to the mill was -120°C. The mill was maintained at -120°C during grinding. 99% of the resulting powder was of a size below 400  $\mu$ m.

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The resultant powder was then applied on to a scrim to a thickness of 5mm by using a Cavistat 91 Pulverstreuaggregat conventional powder scatter head (manufactured by Cavitec). The scrim was a 0.5m width cellulose/polyester support (Dexter 555:030) reinforced with a Kirson '4x4' 34 tex glass crennette moving at a rate of 1 m/minute. The scrim was then passed under a 4 kW medium wave infra red heater (height 5cm above the coated scrim; width 0.6m; length 0.4m). The power output was adjusted in order to partially sinter the powder. Particles of quartz of a size in the range 0.4-0.8 mm were then scattered on to the surface of the powder at a rate of 100 gm<sup>-2</sup>. Particles of silicon carbide size F40 (FEPA Standard 42-GB-1984) were then scattered on to the surface at a rate of 100 gm<sup>-2</sup>. The coated scrim was then passed in to a convection oven where it was exposed to a temperature of 200°C for 3 minutes before being embossed and cooled.

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The product was then tested for slip resistance in wet conditions according to the UK Slip Resistance Guidelines (The measurement of floor slip resistance: Guidelines recommended by the UK slip resistance group ISBN 1-85957-227-8) and shown to have a slip resistance value (SRV) in excess of 40 (coefficient of friction ca 0.4).

## **EXAMPLE 10**

The ingredients of formulation G were weighed and blended by being tumbled together. The blend was extruded in an extruder with a single screw with L:D ratio of 20 and a compression ratio of 2.5:1, maintaining the melt temperature in the region of 180-200°C. The extrudate was water cooled and pelletized. The pellets were dried in a vacuum drier at 50°C and then cryogenically ground in an Alpine pin disc mill using a single pass and no intermediate sieving screen. The temperature of the material prior to its introduction in to the mill was -150°C. The mill was

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maintained at -150°C during grinding. 99% of the resulting powder was of a size below 400  $\mu$ m.

The resultant powder was then applied on to a scrim to a thickness of 5 mm by using a Cavistat 91 Pulverstreuaggregat conventional powder scatter head (manufactured by Cavitec). The scrim was a 0.5 m width cellulose/polyester support (Dexter 555:030) reinforced with a Kirson '4x4' 34 tex glass crennette moving at a rate of 1 m/minute. The scrim was then passed under a 4 kW medium wave infra red heater (height 5 cm above the coated scrim; width 0.6m; length 0.4m). The power output was adjusted in order to partially sinter the powder. Particles of quartz of a size in the range 0.4-0.8 mm were then scattered on to the surface of the powder at a rate of 100 gm<sup>-2</sup>. Particles of silicon carbide size F40 (FEPA Standard 42-GB-1984) were then scattered on to the surface at a rate of 100 g m<sup>-2</sup>. The coated scrim was then passed in to a convection oven where it was exposed to a temperature of 200°C for 3 minutes before being embossed and cooled.

The product was then tested for slip resistance in wet conditions according to the UK Slip Resistance Guidelines (The measurement of floor slip resistance: Guidelines recommended by the UK slip resistance group ISBN 1-85957-227-8) and shown to have a slip resistance value (SRV) in excess of 40 (coefficient of friction ca 0.4).

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The ingredients of formulation B were weighed and blended by being tumbled together. The blend was extruded in a Werner and Pfleiderer extruder (model number ZSK 70) with the feed zone temperature set at 160°C, the barrel set at 190°C and the die at 200°C. The extrudate was water cooled and pelletized. The pellets were dried in a vacuum drier at

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 $40\,^{\circ}\text{C}$  and then cryogenically ground in an Alpine pin disc mill using a single pass and no intermediate sieving screen. The temperature of the material prior to its introduction in to the mill was -120 $^{\circ}\text{C}$ . The mill was maintained at -120 $^{\circ}\text{C}$  during grinding. 99% of the resulting powder was of a size below 400  $\mu\text{m}$ .

The resultant powder of formulation B was then applied on to a scrim to a thickness of 3 mm by using knife over roller. The scrim was a 2m width cellulose/polyester support (Dexter 555:030) reinforced with a Kirson '4x4' 34 tex glass crennette moving at a rate of 3 m/minute. An additional powder layer C (made as per example 4 for formulation A) was then spread on to the first powder layer at an additional thickness of 2 mm using knife over roller. Particles of quartz of a size in the range 0.4-0.8 mm were then scattered on to the surface of the powder at a rate of 100 g/m<sup>2</sup>.

The coated scrim was then passed under a 50 kW medium wave infra red heater (height 10 cm above the coated scrim; width 2.5 m; length 1 m). The power output was adjusted in order to partially sinter the powder. The scrim was then passed in to a convection oven where it was exposed to a temperature of 195°C for 2.5 minutes before being embossed and cooled and wound up for subsequent trimming to size.

The product was then tested for slip resistance in wet conditions according to the UK Slip Resistance Guidelines (The measurement of floor slip resistance: Guidelines Recommended by the UK Slip Resistance Group ISBN 1-85957-227-8) and shown to have a slip resistance value (SRV) in excess of 35 (coefficient of friction ca 0.35).

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### **CLAIMS**

1. A non-laminated plastics floor covering including a first layer of plastics material having an upper surface and a first particulate material embedded in the layer of plastics material and exposed at the upper surface to provide slip resistance, wherein the layer of plastics material is substantially free from a plasticiser.

- A floor covering as defined in Claim 1 which further includes a
   support, preferably a reinforcing support, more preferably a glass fibre reinforced non-woven support.
- 3. A floor covering consisting essentially of a support having an upper surface and a lower surface wherein on the upper surface of the support there is a first layer of plastics material having an upper surface and a first particulate material embedded in the first layer of plastics material and exposed at the upper surface of the plastics material to provide slip resistance and on the lower surface of the support there is optionally a second layer of plastics material.

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- 4. A floor covering as defined in Claim 3 wherein the support is a reinforcing support, preferably a glass fibre reinforced non-woven support.
- 25 5. A floor covering as defined in any one of the preceding Claims wherein the plastics material is substantially free from PVC.
  - 6. A floor covering as defined in any one of the preceding claims wherein the plastics material is a thermoplastic polymer and/or a thermoplastic elastomer.

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7. A floor covering according to claim 6 wherein the plastics material is a thermoplastic polymer, preferably a thermoplastic polymer such as EVA, a polyethylene-acrylic acid copolymer, a terpolymer, and/or a copolymer of ethylene and methacrylic acid.

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8. A floor covering as defined in any one of the preceding Claims wherein the first particulate material is a grit, preferably it is one or more of a number of types of hard particles including silicon carbide, a silica, aluminium oxide and/or emery.

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- 9. A floor covering as defined in any one of the preceding Claims wherein the plastics material contains an ionomer and/or a foaming agent.
- 10. A floor covering as defined in any one of the preceding Claims15 wherein the first layer of plastics material has at least an upper part and a lower part wherein a property of each of the at least two parts of the first layer is different.
- 11. A floor covering as defined in Claim 10 wherein the property is wear resistance, flexibility, scratch resistance, and/or surface hardness.
  - 12. A floor covering as defined in Claim 10 or Claim 11 wherein the upper part of the first layer contains an ionomer, preferably the upper surface of the first layer contains an ionomer.

- 13. A floor covering as defined in any one of Claims 10 to 12 wherein the lower part of the first layer contains a foaming agent.
- 14. A floor covering as defined in any one of Claims 10 to 13 wherein30 the lower part of the first layer contains a second particulate material.

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15. A method of making a slip-resistant floor covering, the method including the consecutive steps of:-

applying a first layer of a plastics material which is substantially free from a plasticiser;

applying a first particulate material to the first layer; melting or curing the plastics material.

16. A method as defined in Claim 15 which is a method of making a floor covering as defined in any one of Claims 1 to 14.

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- 17. A method as defined in Claim 15 or Claim 16 wherein the layer of plastics material is applied to a belt from which the floor covering made by the method can be removed.
- 18. A method as defined in Claim 15 or Claim 16 wherein the floor covering includes a support having an upper surface and a lower surface and wherein the first layer is applied to the upper surface of the support, preferably the support is a reinforcing support, more preferably the support is a glass fibre reinforced non-woven support.

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19. A method as defined in Claim 18 wherein the method includes a first or subsequent step of applying a second layer of plastics material which is substantially free from plasticiser to the lower surface of the support.

- 20. A method as defined in any one of Claims 15 to 19 wherein the plastics material includes an ionomer and/or a foaming agent.
- 21. A method as defined in any one of Claims 15 to 20 wherein the 30 first and/or second layer of plastics material is applied in two or more

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stages such that the composition and/or properties of the plastics material in the layer can be varied.

- 22. A method as defined in Claim 21 which further includes one or more steps of cohering the layer of plastics material after application of one stage and before a further stage is applied to reduce scattering of the plastics material and mixing between the different stages.
- 23. A method as defined in Claim 22 wherein the cohering step includes partially sintering the particles of the plastics material.
  - 24. A method as defined in Claim 22 or Claim 23 wherein the plastics material is cooled after the cohering step and before the application of a further stage.

- 25. A method as defined in any one of Claims 21 to 24 wherein the plastics material applied in the second or subsequent stage includes an ionomer.
- 20 26. A method as defined in Claim 25 wherein the ionomer is applied in two parts with a first part being applied in the second or subsequent stage and a second part being applied in a third or further stage.
- 27. A method as defined in Claim 26 wherein the first part of the ionomer is a compound including a metal cation and the second part of the ionomer is a copolymer of an olefin monomer and a polymerisable carboxylic acid monomer or the first part of the ionomer is a copolymer of an olefin monomer and a polymerisable carboxylic acid monomer and the second part of the ionomer is a compound including a metal cation.

28. A method as defined in Claim 27 wherein the first part of the ionomer is a copolymer of an olefin monomer and a polymerisable carboxylic acid monomer and the second part of the ionomer is a compound including a metal cation.

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29. A method as defined in any one of Claims 21 to 28 wherein the plastics material applied in the first stage includes a foaming agent, preferably such that the lower part of the layer of plastics material is flexible.

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- 30. A method as defined in any one of Claims 21 to 29 wherein the first layer of plastics material is formed from two or more stages and the first particulate material penetrates the first stage of the layer.
- 15 31. A method as defined in any of Claims 15 to 30 wherein before the step of applying the first particulate material, the method includes a step of cohering the plastics material.
- 32. A method as defined in any of Claims 15 to 30 wherein the first particulate material is applied in a manner such that its kinetic energy on impact with the plastics material is minimised.
  - 33. A method as defined in Claim 32 wherein after the step of applying the first particulate material, the plastics material is sintered.

- 34. A method as defined in any one of Claims 15 to 33 wherein the plastics material is applied in the form of a powder, granules or pellets, preferably in the form of a powder.
- 30 35. A method as defined in Claim 34 wherein the plastics material is applied to the desired thickness using knife over roller or knife over air.

- 36. A method as defined in Claim 35 wherein the plastics material is a thermoplastic plastics material, preferably a thermoplastic elastomer.
- 5 37. A floor covering obtainable by a method as defined in any one of Claims 15 to 36.
  - 38. A floor covering as defined in any one of Claims 1 to 14 obtainable by a method as defined in any one of Claims 15 to 36.
  - 39. A floor covering substantially as hereinbefore described with reference to Figure 2 of the accompanying drawings.

- 40. A method substantially as hereinbefore described with reference to Figure 1 of the accompanying drawings.
  - 41. A method of making a floor covering including a layer of plastics material wherein a portion of the plastics material includes an ionomer which method includes the steps of
- applying at least a part of a layer of plastics material including a copolymer of an olefin monomer and a polymerisable carboxylic acid monomer;
  - applying a compound including a metal cation to the surface of the part of the layer of plastics material;
- curing the plastics material such that the metal cation at least partially neutralises the carboxylic acid monomer.

FIGURE 1

