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(54) Titre : PIGMENTS SOUS FORME DE GRANULES POUR TEINTER DES SUBSTANCES HOMOPOLAIRES ET PROCEDE PERMETTANT DE LES PRODUIRE

(54) Title: PIGMENT GRANULATE FOR THE COLOURING OF HOMOPOLAR MEDIA AND PROCESSES FOR ITS MANUFACTURE

(57) Abrégé/Abstract:
The invention pertains to pigment granulates for the colouring of asphalt, bitumen, bituminous materials, tar, and plastics as well as processes to manufacture such granulates. The mixture to be granulated consists of a mixture containing pigments, at least one agent to promote the colouring and distribution in homopolar media and/or at least one dispergator for polar systems as well as solvents if necessary.

Abstract

The invention pertains to pigment granulates for the colouring of asphalt, bitumen, bituminous materials, tar, and plastics as well as processes to manufacture such granulates.

The mixture to be granulated consists of a mixture containing pigments, at least one agent to promote the colouring and distribution in homopolar media and/or at least one dispergator for polar systems as well as solvents if necessary.

Pigment Granulate for the Colouring of Homopolar Media and Processes for Its Manufacture

The invention pertains to pigment granulate for the colouring of homopolar media, like asphalt, bitumen, bituminous materials, tar, and plastics, and processes for the manufacture of such granulates.

At present, in addition to organic pigments, inorganic pigments, especially those based on different iron oxides, are also used to colour homopolar materials, especially asphalt and plastics. Compared to powders and pastes, pigment granulates exhibit very considerable advantages in this regard, for example in their handling, their prevention of dust and the like, but also in regard to their dispersing properties.

The use of aqueous carbon preparations (containing 30 to 80% water) as pearl granulates for colouring in the cement industry is known from DE-Al 29 08 202. This process cannot be applied to other pigments, and granulates with such water contents are fundamentally disadvantageous, also for applications involving asphalt and plastics.

The granulation of pigments together with binders through spray granulation is known from DE-Al 29 40 156. Pigment granulates produced in this way are used to manufacture inks, to colour plastics, lacquers, and the like. There is no connection to the colouring of building materials, asphalt, and the like.

Colour granulates for building materials containing more than 5 to 50% by weight of water are known from EP-A2 0 191 278.

EP-Al 0 567 882 describes pelletized, compacted, and sprayed granulates containing binders, such as especially machine oil, wax, paraffin, and the like, which are to be used to colour asphalt, among other things. In addition to the binders, substances like lignin sulfonate, molasses, starch, and the like can be used. According to this disclosure, substances like lignin sulfonate alone cannot be used as binders.

In the context of this description, "granulates" means every material whose mean grain size in comparison to the original material is increased by a treatment stage. Therefore, "granulates" means not only spray granulates and compacting granulates but, for example, also products of a moist treatment with subsequent pulverizing.

On account of their considerable advantages, in comparison to powders, pastes, and the like, granulates have been used for decades on a large scale industrially. Granulation has been accepted for a long time for the processing of pigments as well.

Experience acquired with pigment granulates in other areas cannot simply be transferred to the colouring of building materials, asphalt, and the like. In practice, granulates, which theoretically should be almost perfectly suitable, often prove inadequate because they do not combine all of the required properties.

Although often an excellent solidity of the granulate is achieved, which counteracts its destruction during packaging and transport and restricts the production of dust, on the other hand, its dispersability can be impaired, resulting in the desired homogeneous colouring and the required colour intensity not being achieved.

On the other hand, granulates with excellent colour intensity and easy dispersability are often too soft and already disintegrate before they are worked into the asphalt or plastic, which can result in increased dust production, in residues in the packaging, in reduced flowability and in corresponding frequent incorrect dosages.

Of the various methods of granulation, spray granulation has become generally accepted in practice, while, for example, fluidized bed granulations, which is fully comparable theoretically, has not yet produced any usable granulates.

Proposals have recently become known to forego granulates completely and, instead, to use coated powders.

WO 97/20892 is given as an example. It remains to be seen whether in this way broad particle size distributions due to clumping can be avoided and whether such coated powders can be used without producing the dust problems typical of the prior art before pigment granulates were introduced.

In order to promote the dispersion and distribution of pigment granulates, wetting and binding agents are used which are chosen in such a way that the granulate disintegrates with the desired dispersing effect. For the colouring of homopolar media, such as especially asphalt and plastics, hydrophobic compounds, like oils and waxes, which guarantee an adequate dispersability of the granulates in the hydrophobic application medium, are traditionally used as binders for the mixture to be granulated. The disadvantages of mixtures to be granulated which are based on homopolar solvents are, first, the high costs in comparison to a mixture based on water, for example, and, second, the fact that special technical devices and security measures are required for the evaporating organic binders, like oils and waxes where granulates are spray dried.

Water-based wetting agent and binder mixtures, for which water-soluble wetting agents and binders, like lignin sulfonate and the like, exhibit the disadvantage, however, the pigment cannot be homogeneously distributed in a homopolar application medium, like asphalt, bitumen, or plastic. This result in an uneven colouring, which is undesirable.

Therefore, an essential objective of the invention is to propose, against this background, a process for the colouring of homopolar media, like asphalt, bitumen, bituminous materials, tar, and plastics, by means of pigment granulates which promotes the colouring by the pigments and at the same time improves the dispersion of the pigment in a homopolar application medium. Another objective of the invention is to present a process which promotes the sprayability of the granulate.

The objective of the invention is solved by the pigments, in particular iron oxides and/or soot pigments, and the traditional wetting and binding agents, also being mixed during the manufacture of the pigment granulates with at least one agent which promotes the colouring and the distribution of the pigment in homopolar media and/or at least one dispersing agent for polar systems. Surprisingly, it was found that the agents to promote the colouring and the distribution of the pigment in homopolar media in accordance with this invention act in such a way that a lipophilic coating is produced on the granulate as a result of which the dispersion and, therefore, the homogeneous distribution of the pigment in homopolar media, like asphalt, bitumen, bituminous materials, tar, and plastics, is promoted and, consequently, also the colouring of the homopolar media is promoted in an above-average manner.

Such an agent which promotes the colouring and the distribution of the pigment and which can be used to manufacture the inventive pigment granulate is preferably a wax or a mixture of several waxes. When using waxes, attention must be paid to the wax having both a high scratch and abrasion resistance. In a preferred embodiment of the invention, waxes with these properties have a melting point in the 50 to 200°C range, preferably 50 to 130°C.

It is irrelevant with this invention whether the waxes are natural or synthetic in origin. Preferably, however, synthetic waxes are used, like polyalkylene waxes, especially polyethylene waxes, polyethylene glycol waxes, paraffin waxes, styrene acrylate waxes, polytetrafluoroethylene waxes, and the like.

With this invention, non-ionogenic waxes, waxes with anionic ionogenity, waxes with cationic ionogenity and combinations of these waxes are used. Preferably, wax mixtures are used, in which case both mixtures of waxes with anionic and/or cationic ionogenity or mixtures of non-ionogenic waxes as well as mixtures of non-ionogenic waxes and waxes with anionic or cationic ionogenity can be used. Especially preferred, mixtures of polyethylene wax and styrene-acrylate wax or mixtures of polyethylene wax and paraffin wax can be used.

In order to increase the solid contents, especially of the pigment contents, in the mixture to be granulated, in accordance with the invention, dispersing agents or dispergators for polar systems can be used which promote the liquefication of solid pigment after polar solvents are added, such as water in particular, and the shaping of the granulates, especially with spray drying. The latter is especially advantageous if the aforesaid agents which promote the colouring and the distribution of the pigment in homopolar media are contained in the mixture to be granulated. With this preferred embodiment, a synergistic effect results due to the fact that both the pigment contents in the mixture to be granulated and the shaping of the granulates are increased as well as the homogeneity of the colouring in the homopolar medium. The dispergators can also be used advantageously, however, if none of the agents which promote colouring and distribution of the pigment in homopolar media are used because, for example, with spray drying the increase in the pigment fraction in the pigment slurry to be sprayed results in the spray costs being reduced substantially. As well, the agents which promote the colouring and the distribution of the pigment in homopolar media can also be used advantageously if the dispergators for polar systems are not used.

Dispergators within the scope of this invention are defined, as in the Römpp Chemie Lexikon [Römpp Chemistry Lexicon], Georg Thieme Verlag [Georg Thieme Publishers], Stuttgart, New York, ninth edition, 1990, page 1010, as agents which facilitate the dispersing of particles in a dispersion agent by lowering the surface tension between the two components.

With this invention the dispergators for polar systems in the mixture to be granulated can be chosen from hydrophilic and amphoteric, ionogenic, and non-ionogenic compounds. Preferably, these agents can be chosen from mono- or polyhydroxy compounds, mono- or polyhydroxy amine compounds, (poly) carboxylates, polyacrylates, lignin sulfonates, sulfated polyglycol ethers, melamine formaldehyde condensates, naphthalene formaldehyde condensates, alkyl-, aryl, or alkylaryl sulfonates, polyglycols, polyglycol derivatives, PVP, polyethers, phosphates, silicates, aluminates, borates, cellulose derivatives, or combinations of these compounds.

Monovalent, primary, secondary, or tertiary, alkyl-substituted or non-substituted alcohols, such as, for example, 1-propanol, 2-methyl-1-propanol, 2-methyl-2-propanol, and the like can be used as monohydroxy compounds. Alcohols from C3 up are preferred. In accordance with the invention, the polyhydroxy compounds used include polyhydric alkyl-substituted or non-substituted alcohols, for example diols, glycols, like ethylene glycol and polyalkylene glycol, glycerine, sugar alcohols, like sorbitol and ionosite, trimethylol propane, and the like. Preferably, 2-methyl-1-propanol and glycols are used in the case of this invention.

The monohydroxyamino compounds used as dispergators include monovalent, primary, secondary, or tertiary, alkyl-substituted or non-substituted amino alcohols, such as 2-amino-1-propanol, 2-amino-1-butanol, 3-amino-1-propanol, 2-amino-2-methyl-1-propanol, and the like. Amino alcohols from C_3 up are preferred. Polyhydric alkyl-substituted or non-substituted alcohols, such as, for example, 2-amino-2-methyl-1,3-dihydroxy propane,

and the like can be used as inventive polyhydroxy amine compounds. In a preferred embodiment of this invention, 2-amino-2-methyl-l-propanol is used.

Other compounds with relatively low molecular weights (preferably C_1 to C_{100}, especially preferred C_2 to C_{50} especially C_3 to C_{25}), which carry one or more hydrophilic groups, can also be used. These can be NH_2 and OH, but also ether groups, carboxylic groups, acid functions, and the like, if necessary also in neutralized (salt) form.

The total quantity of the agents to promote the colouring and the distribution of the pigment in homopolar media used in the inventive process for the manufacture of pigment granulates is at least 0.01 percent by weight, preferably 0.01 to 5 percent by weight, and especially preferred 0.4 to 3.5 percent by weight, based on the total quantity of the mixture to be granulated.

The total quantity of the dispergators for polar systems used in the inventive process for the manufacture of pigment granulates is at least 0.05 percent by weight, preferably 0.1 to 3 percent by weight, and especially preferred 0.25 to 1.7 percent by weight, based on the total quantity of the mixture to be granulated.

In accordance with the invention, the pigments can be mixed as a powder mixture or suspended in a solvent with at least one agent to promote the colouring and the distribution in homopolar media and/or at least one dispergator for polar systems. The resulting mixtures can be produced through compression, compacting, pressing, or briquetting, spraying, fluidized bed drying, or through pelletizing, or combinations of the aforesaid processes. Preferably, spray processes are used.

The use of granulates during mixing with homopolar media, like asphalt, bitumen, bituminous materials, tar, and plastics, corresponds to the customary, known procedures.

The following examples are intended to explain the invention.

Examples 1 to 4

Four pigment granulates suitable for the inventive process for the colouring of homopolar media, especially asphalt and plastics, were manufactured in accordance with the recipes presented in the following tables:

Pigment granulate mixture 1

Substance	Percent by weight
Fresh water	34.66
Polyacrylate	1.15
2-amino-2-methyl-1-propanol	0.25
Iron oxide red	62.50
Sodium hydroxide solution (20%)	0.20
Wükonil™ LP 50	0.83
Südranol™ 340	0.31
Wükonil™ MS 30	0.10

Pigment granulate mixture 2

Substance	Percent by weight
Fresh water	35.31
Lignin sulfonate	0.5
2-amino-2-methyl-1-propanol	0.25
Iron oxide red	62.50
Sodium hydroxide solution (20%)	0.20
Ultralube™ E 340	0.83
Ultralube™ MD 2000	0.41

Pigment granulate mixture 3

Substance	Percent by weight
Fresh water	32.5
Polyethylene propylene glycol	1.5
2-amino-2-methyl-1-propanol	0.25
Iron oxide red	62.5
Sodium hydroxide solution (20%)	0.20
Wükonil™ LP 50	1.66
Südranol™ 340	0.89

Wükonil™ MS 30	0.50

Pigment granulate mixture 4

Substance	Percent by weight
Fresh water	33.42
Polyacrylate	1.15
2-amino-2-methyl-1-propanol	0.25
Iron oxide red	62.50
Sodium hydroxide solution (20%)	0.20
Ultralube™ E 340	1.66
Ultralube™ MD 2000	0.82

The brand names in the above tables represent the following:

Wükonil™ LP 50:	macroparaffin (anionic, melting range: 60°C)
Südranol™ 340:	polyethylene wax (anionic, melting range: 95°C)
Wükonil™ MS 30:	styrene-acrylate wax (anionic, melting range: > 80°C)
Ultralube™ MD 2000:	polyethylene wax (non-ionogenic, melting range: l27°C)
Ultralube™ E 340:	paraffin wax (anionic, melting range: 56°C - 58°C)

THE EMBODIMENTS OF THE INVENTION IN WHICH AN EXCLUSIVE PROPERTY OR PRIVILEGE IS CLAIMED ARE DEFINED AS FOLLOWS:

1. A pigment granulate for the colouring of homopolar media, selected from asphalt, bitumen, bituminous materials, tar and plastics, prepared from a mixture comprising pigments, mixtures of non-ionogenic waxes and/or waxes with anionic ionogenity and/or waxes with cationic ionogenity, and at least one dispersant for polar systems and optionally solvent.

2. The granulate of claim 1, wherein the waxes used have a melting point in the range of 50°C to 200°C.

3. The granulate of claim 2, wherein the waxes used have a melting point in the 50°C to 130°C range.

4. The granulate of any one of claims 1 to 3, wherein the waxes used contain a mixture of polyethylene wax and styrene-acrylate wax.

5. The granulate of any one of claims 1 to 4, wherein the waxes used contain a mixture of polyethylene wax and paraffin wax.

6. The granulate of any one of claims 1 to 5, wherein the total quantity of waxes ranges from 0.1 to 5% by weight based on the total weight of the mixture to be granulated.

7. The granulate of any one of claims 1 to 5, wherein the total quantity of waxes ranges from 0.4 to 3.5% by weight based on the total weight of the mixture to be granulated.

8. The granulate of claim 1, wherein the dispersant for polar systems is selected from the group consisting of mono- or polyhydroxy compounds, mono- or polyhydroxyamine compounds, (poly)carboxylates, polyacrylates, lignin sulfonate, sulfated polyglycol ethers, melamine formaldehyde condensates, naphthalene formaldehyde condensates, alkyl-, aryl, or alkylaryl sulfonates, polyglycols, polyglycol derivatives, polyethers, cellulose derivatives, and combinations of these compounds.

9. The granulate of claim 8, wherein the hydroxyamine compounds comprise aminoethylpropanols.

10. The granulate of claims 8 or 9, wherein the hydroxy compounds comprise methyl propanols and glycols.

11. The granulate of claim 1, wherein the total quantity of the dispersants for polar systems ranges from 0.1 to 3% by weight based on the total weight of the mixture to be granulated.

12. The granulate of claim 1, wherein the total quantity of the dispersants for polar systems ranging from 0.25 to 1.7% by weight based on the total weight of the mixture to be granulated.

13. The granulate of any one of claims 1 to 12, wherein the pigment is selected from the group consisting of an iron oxide pigment and a soot pigment.

14. A granulate according to any one of claims 1 to 13, characterized in that the solvent is a polar solvent.

15. A granulate according to claim 14, wherein the polar solvent is water.

16. A process to manufacture a granulate according to any one of claims 1 to 14,

characterized in that the pigments in the form of a powder mixture or a suspension in a solvent are blended with mixtures of non-ionogenic waxes and/or waxes with anionic ionogenity and/or waxes with cationic ionogenity and at least one dispersant for polar systems, and the resulting mixture is granulated by compressing, compacting, pressing, briquetting, spraying, fluidized bed drying or pelletizing or combinations of the above-mentioned granulating methods.

17. A process according to claim 16,

characterized in that the granulate is manufactured with the use of spraying methods.