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(54) Title: INTERCALATION AGENT FREE COMPOSITIONS USEFUL TO MAKE NANOCOMPOSITE POLYMERS

(57) Abstract: A two step method for preparing a filler composition, the filler composition useful to prepare a nanocomposite polymer. The first step is to disperse a water dispersible filler material in a liquid comprising water to form a dispersion. The second step is to replace at least a portion of the water of the liquid with an organic solvent to form the filler composition, the water concentration of the liquid of the filler composition being less than six percent by weight, the average size of at least one dimension of the filler material being less than two hundred nanometers upon examination by transmission electron microscopy of a representative freeze dried sample of the dispersion of the first step. A nanocomposite polymer can be prepared by mixing the above-made filler composition with one or more polymer, polymer component, monomer or prepolymer to produce a polymer containing a filler having an average size of at least one dimension of the filler of less than two hundred nanometers upon examination by transmission electron microscopy of a representative sample of the polymer. In addition, an epoxy resin composition useful for making a cured epoxy nanocomposite polymer, the epoxy resin composition made by a two step process. The first step is to mix an epoxy resin with the filler composition to form an epoxy resin mixture. The second step is to remove organic solvent from the epoxy resin mixture to form the epoxy resin composition. And, a nanocomposite polymer, made by the step of mixing the filler composition with one or more polymer, polymer component, monomer or prepolymer to produce a polymer containing a filler having an average size of at least one dimension of the filler of less than two hundred nanometers upon examination by transmission electron microscopy of a representative sample of the polymer.

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## A. CLASSIFICATION OF SUBJECT MATTER

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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)

C08J C08K

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, PAJ, WPI Data

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

| Category* | Citation of document, with indication, where appropriate, of the relevant passages                                                           | Relevant to claim No. |
|-----------|----------------------------------------------------------------------------------------------------------------------------------------------|-----------------------|
| E         | WO 2007/133763 A2 (DOW GLOBAL TECHNOLOGIES INC [US]; DINEGA DMITRY P [US])<br>22 November 2007 (2007-11-22)<br>abstract; claims 1-27         | 1-18                  |
| X         | US 2002/098309 A1 (BAGRODIA SHRIRAM [US] ET AL) 25 July 2002 (2002-07-25)<br>abstract; claims 1-26<br>page 8, paragraph 100<br>-----<br>-/-- | 1-12,<br>14-15, 18    |

☒ Further documents are listed in the continuation of Box C.☒ See patent family annex.

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C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

| Category* | Citation of document, with indication, where appropriate, of the relevant passages                                                                                                                                                                                                                                                                                           | Relevant to claim No. |
|-----------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------|
| X         | LEI WANG ET AL: "Hydrothermal effects on the thermomechanical properties of high performance epoxy/clay nanocomposites"<br>POLYMER ENGINEERING & SCIENCE, WILEY, HOBOKEN, NJ, US,<br>vol. 46, no. 2,<br>1 February 2006 (2006-02-01), pages 215-221, XP002466055<br>ISSN: 0032-3888<br>the whole document<br>-----                                                           | 1-12,18               |
| X         | CHEN C ET AL: "ORGANOCLAY-AEROSPACE EPOXY NANOCOMPOSITES"<br>INTERNATIONAL SAMPE SYMPOSIUM AND EXHIBITION, XX, XX,<br>vol. 1, 6 May 2001 (2001-05-06), pages 362-374, XP008051803<br>the whole document<br>-----                                                                                                                                                             | 1,13-18               |
| X         | CHEN BIN ET AL: "Synthesis of disordered and highly exfoliated epoxy/clay nanocomposites using organoclay with catalytic function via acetone-clay slurry method"<br>CHEMISTRY OF MATERIALS, AMERICAN CHEMICAL SOCIETY, WASHINGTON, US,<br>vol. 16, no. 24,<br>30 November 2004 (2004-11-30), pages 4864-4866, XP002465720<br>ISSN: 0897-4756<br>the whole document<br>----- | 1,18                  |
| X         | WANG KE ET AL: "Epoxy nanocomposites with highly exfoliated clay: Mechanical properties and fracture mechanisms"<br>MACROMOLECULES, ACS, WASHINGTON, DC, US,<br>vol. 38, no. 3,<br>8 February 2005 (2005-02-08), pages 788-800, XP002466054<br>ISSN: 0024-9297<br>the whole document<br>-----                                                                                | 1,13,18               |

# INTERNATIONAL SEARCH REPORT

Information on patent family members

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| Patent document<br>cited in search report | Publication<br>date | Patent family<br>member(s) | Publication<br>date |
|-------------------------------------------|---------------------|----------------------------|---------------------|
| WO 2007133763                             | A2                  | 22-11-2007                 | CA                  |
|                                           |                     | EP                         | 2649979 A1          |
|                                           |                     |                            | 2021284 A2          |
|                                           |                     |                            | 22-11-2007          |
|                                           |                     |                            | 11-02-2009          |
| US 2002098309                             | A1                  | 25-07-2002                 | NONE                |