"as originally filed"

METHOD FOR PRODUCING ISOSORBIDE ETHOXYLATE DIMETHACRYLATE

Description

The invention relates to a process for preparing isosorbide ethoxylate di(meth)acrylate by transesterifying alkyl (meth)acrylate with isosorbide ethoxylate, to the isosorbide ethoxylate di(meth)acrylate itself and to the use thereof.

E2BADMA (dimethacrylate of bisphenol A alkoxylated with 2 ethylene oxide units in each case) and E3BADMA (dimethacrylate of bisphenol A alkoxylated with 3 ethylene oxide units in each case) are bisphenol A-based dimethacrylates which are used as crosslinkers, for example in coatings and mixtures for drillholes. They are notable for a rapid reaction time which leads to partial load-bearing capacity of 2-pack mixtures even after a short time and attainment of the final tensile strength after a few hours. In addition, they have a high polymer density with a very good cohesion/adhesion ratio, good crystallization resistance with simultaneously low shrinkage and a low tendency to embrittlement, and high wetting power in order to assure good creeping capacity in capillary cracks.

DE 41 31 458 A1 discloses two-pack adhesive compositions for chemical securing technology, comprising a synthetic resin comprising the di(meth)acrylate of an alkoxylated bisphenol and a hardener for the synthetic resin.

For particular applications, bisphenol A-free crosslinkers having a similar profile of properties are being sought.

Isosorbide is a diol based on renewable raw materials and, in structural terms, is an alternative to bisphenol A, since it has a similarly rigid base structure.

One means of preparing di(meth)acrylates from isosorbide is the esterification of isosorbide with (meth)acrylic acid or the transesterification of methyl acrylate, ethyl acrylate or methyl methacrylate with isosorbide in the presence of suitable catalysts. Both OH groups of isosorbide are secondary OH groups having comparatively low reactivity, and the relative reactivities of the two OH groups are additionally very different. Thus, incomplete conversion is to be expected.

DE 2 317 226 A1 discloses a method of preparing (meth)acrylic esters from a mixture of C_{10}-C_{18} alkanols by transesterification of methyl (meth)acrylate in the presence of titanium alkoxide as catalyst and 2,6-di-tert-butylparacresol (TBC) as stabilizer. This method is carried out in the presence of activated carbon. Once the reaction has ended, water is added to hydrolyze the titanium alkoxide to titanium hydroxide/oxide which adsorbs onto the activated carbon. The solid is filtered off and the reaction product is subjected to a steam distillation.

Claims

1. A process for preparing isosorbide ethoxylate di(meth)acrylate by transesterifying alkyl (meth)acrylate with isosorbide ethoxylate, comprising the steps of:

   (i) ethoxylating isosorbide to give isosorbide ethoxylate,
   (ii) reacting alkyl (meth)acrylate with isosorbide ethoxylate in the presence of potassium phosphate as catalyst and a stabilizer and in the presence of an azeotroping agent which forms an azeotrope with the alcohol bound in the alkyl (meth)acrylate,
   (iii) continuously distilling off the azeotrope of azeotroping agent and alcohol, wherein steps (ii) and (iii) are conducted simultaneously until the isosorbide ethoxylate has been essentially fully converted,
   (iv) removing the catalyst from the product mixture comprising isosorbide ethoxylate di(meth)acrylate,
   (v) distilling unconverted alkyl (meth)acrylate and azeotroping agent out of the product mixture.

2. The process according to claim 1, wherein the azeotroping agent is the alkyl (meth)acrylate.

3. The process according to claim 1, wherein the azeotroping agent is a separate solvent other than alkyl (meth)acrylate.

4. The process according to claim 3, wherein the azeotroping agent is selected from the group consisting of n-heptane and cyclohexane.

5. The process according to any of claims 1 to 4, wherein the alkyl (meth)acrylate is methyl or ethyl (meth)acrylate.

6. The process according to any of claims 1 to 5, wherein the stabilizer is hydroquinone monomethyl ether.

7. The process according to any of claims 1 to 6, wherein the alcohol is removed from the azeotrope of entraining agent and alcohol distilled off in step (iii) by washing with water and the azeotroping agent is recycled into the reaction vessel.

8. The process according to any of claims 1 to 7, wherein, after step (v), an isosorbide ethoxylate di(meth)acrylate having a content of secondary components of < 4% by weight is obtained.

9. The process according to claim 8, wherein, after step (v), an isosorbide ethoxylate di(meth)acrylate having a content of secondary components of < 2% by weight is obtained.

10. The use of isosorbide ethoxylate di(meth)acrylate as resin component for two-pack adhesive compositions.

11. The use according to claim 10, wherein the two-pack adhesive compositions are plugging compounds for securing anchoring means in drillholes.

12. The use according to claim 10 or 11, wherein the isosorbide ethoxylate di(meth)acrylate has been prepared by the process according to any of claims 1 to 9.

13. A two-pack adhesive composition comprising isosorbide ethoxylate di(meth)acrylate as resin component.