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Fortsættes ...

Description

The invention relates to a method of producing rigid polyurethane foams and foamed thermoplastic polymers.

Polyurethane foams are employed as building products having a heat insulation and/or noise reduction effect. Production of single- and multi-component rigid polyurethane foams with blowing agents based on liquefied CO2 is disclosed in WO 96/14354.

Foamed thermoplastic polymers can be employed, for example in the form of panels, as a thermally and/or acoustically insulating component part. US-A 5,276,063 discloses a method of producing extruded closed-cell alkenylaromatic polymers by using a blowing agent mixture which provides 1,1-difluoroethane and also a further blowing agent of lower vapour pressure and still higher solubility in the molten polymer. Suitable alkenylaromatic polymers are, for example, polymers of styrene, alpha-methylstyrene, ethylstyrene, vinylbenzene, vinyltoluene, chlorostyrene and bromostyrene. These polymers may optionally include copolymers such as acrylic acid, acrylonitrile or butadiene. US-A 5,204,169 discloses the production of foamed thermoplastic polymers such as polystyrene by use of polyfluorinated hydrocarbons having 2 carbon atoms. The foamed material is particularly useful for food packaging. EP-A-0 436 847 discloses the production of foamed thermoplastic articles based on polyphenylene ether resins. Hydrocarbons are recommended as blowing agents. Halogenated hydrocarbons having 1 or 2 carbon atoms are also mentioned as usable.

The problem addressed by the present invention is that of devising a method of producing rigid polyurethane foams via a selected, novel, advantageous blowing agent. The problem addressed by the present invention is further that of devising a method of producing foamed thermoplastic polymers via a novel, advantageous blowing agent. These problems are solved by the method and the blowing agents of the present invention.

The starting point was the surprising realization that pentafluorobutane, particularly 1,1,1,3,3-pentafluorobutane (HFC-365mfc), mixes with certain further blowing agents into a very highly suitable composition for the production of rigid polyurethane foams and foamed thermoplastic polymers.

One method of producing rigid polyurethane foams and foamed thermoplastic polymers by means of a blowing agent provides that the blowing agent used is a composition containing or consisting of a) pentafluorobutane, preferably 1,1,1,3,3-pentafluorobutane (HFC-365mfc), and b) at least one further blowing agent selected from the group comprising low-boiling aliphatic hydrocarbons, ethers and haloethers; difluoromethane (HFC-32); difluoroethane, preferably 1,1-difluoroethane (HFC-152a); 1,1,2,2-tetrafluoroethane (HFC-134); 1,1,1,2-tetrafluoroethane (HFC-134a); pentafluoropropane, preferably 1,1,1,3,3-pentafluoropropane (HFC-245fa); hexafluoropropane, preferably 1,1,2,3,3,3-hexafluoropropane (HFC-236ea) or 1,1,1,3,3,3-hexafluoropropane (HFC-236fa); and heptafluoropropane, preferably 1,1,1,2,3,3,3-heptafluoropropane (HFC-227ea).

The blowing agent used in the present invention consists of 10 to < 50 wt% of 1,1,1,3,3-pentafluorobutane and > 50 wt% of 1,1,1,3,3-pentafluoropropane.

The term “low-boiling hydrocarbons, ethers and haloethers” is to be understood as meaning compounds having a below boiling point below 70 DEG C, preferably below 55 DEG C. Suitable hydrocarbons are hydrocarbons having 2 to 6 carbon atoms, e.g. ethane, propane, butane, pentane and hexane and also mixtures thereof. Isomerically pure compounds or mixtures of various isomers may be employed here. “Butane” is to be understood as meaning mixtures of n-butane and i-butane. Mixtures of this type are commercially available. Pure n-butane or i-butane or their mixture in any desired composition is also usable, but very costly. The same applies to higher homologues such as pentane, etc.

Also highly useful for application in the method are blowing agent compositions which in addition to HFC-365mfc and one or more of the blowing agents recited above under b), such as hydrocarbons or hydrofluorocarbons, also contain liquefied carbon dioxide. The amount of CO_2 in the blowing agent composition is then preferably from 2 to 50 wt%. The blowing agent composition may additionally further contain up to 30 wt% of modifiers for the properties of the polymer to be produced.

Very highly suitable blowing agents are, for example, the following compositions, which may contain or consist of (examples of compositions with parts by weight indicated between parentheses):

HFC-365mfc and HFC-152a (70:30);
HFC-365mfc and HFC-32 (70:30);
HFC-365mfc, HFC-152a and CO_2 (60:30:10);
HFC-365mfc, HFC-32 and CO_2 (60:30:10);
HFC-365mfc, HFC-152a and butane (60:30:10);
HFC-365mfc, HFC-32 and butane (60:30:10);
HFC-365mfc, HFC-152a and HFC-134a (60:25:15);
HFC-365mfc, HFC-32 and HFC-134a (60:25:15);
HFC-365mfc and dimethyl ether (80:20);
HFC-365mfc and pentane (50:50);
HFC-365mfc and propane (70:30);
HFC-365mfc and ethane (90:10);
HFC-365mfc, pentane and CO_2 (45:45:10);
HFC-365mfc, butane and CO_2 (50:40:10);
HFC-365mfc, propane and CO_2 (70:20:10);
HFC-365mfc, ethane and CO_2 (90:5:5).

Other blowing agent compositions contain 1,1,1,3,3-pentafluorobutane and difluoromethane and/or 1,1-difluoroethane, or they consist of these compounds. Very particular emphasis is given to employing compositions containing or consisting of 10 to 70 wt% of HFC-365mfc and 90 to 30 wt% of HFC-152a and/or HFC-32.

The production of rigid PU foams will now be more particularly described.

Outstandingly usable flameproofing agents include, for example, reactive flameproofing agents such as brominated polyols. Likewise suitable are flameproofing agents based on organophosphorus compounds, for example phosphate esters and phosphonoesters. These have organic groups, which may also be substituted by one or more halogen atoms. The organic groups may be aliphatic or aromatic in character. Very highly suitable are phosphate esters and phosphonate esters substituted by three C1-C6 alkyl groups, which may have one or two halogen atoms, examples being trischloroisopropyl phosphate, trischloroethyl phosphate, trischloropropyl phosphate, dimethyl ethyl phosphate, trisdichloroisopropyl phosphate, dimethyl methylphosphonate, preferably trischloropropyl phosphate.

One method of producing rigid polyurethane foams provides that when a) HFC-365mfc and b) 1,1,1,2-tetrafluoroethane (HFC-134a); 1,1,1,3,3-pentafluoropropane (HFC-245fa); 1,1,1,3,3,3-hexafluoropropane (HFC-236fa); or 1,1,1,2,3,3,3-heptafluoropropane (HFC-227ea), but no CO2, low-boiling, optionally halogenated hydrocarbons, ethers or halogenated ethers are present, the blowing agent composition contains or consists of less than 50 wt% of 1,1,1,3,3-pentafluorobutane and more than 50 wt% of 1,1,1,2-tetrafluoroethane; 1,1,1,3,3-pentafluoropropane; 1,1,1,3,3,3-hexafluoropropane or 1,1,1,2,3,3,3-heptafluoropropane.

The effective amount of the blowing agent composition employed in the method can be determined in simple tests carried out by hand. The blowing agent composition is advantageously employed in an amount of 1 to 50 wt%, based on the combined mixture of polymer to be foamed and/or the precursors (polyol, isocyanate, auxiliaries) as well as the blowing agent composition.

Substantially closed-cell rigid polyurethane foams characterized by the presence in the cells of the blowing agent composition to be applied in the method of the present invention are obtainable.

The production of such foams and the foundationstock materials usable therefor and the manner of foaming are disclosed in European patent application EP-A-0 381 986; in "Ullmann's Encyclopedia of Industrial Chemistry", 5th edition, volume A21, pages 665-680; the international patent applications WO 92/00345, 96/30439, 96/14354 and German *Offenlegungsschrift* DE 44 22 714 A1. Polyisocyanates are used, for example with 2 to 4 isocyanate groups.

They include an aliphatic hydrocarbyl moiety of up to 18 carbon atoms, a cycloaliphatic hydrocarbyl moiety of up to 15 carbon atoms, an aromatic hydrocarbyl moiety of 6 to 15 carbon atoms or an aliphatic hydrocarbyl moiety of 8 to 15 carbon atoms. Industry has a particular preference for starting components such as, for example, diphenylmethane diisocyanate, polymethylene polyphenyl isocyanate and mixtures thereof. Also employable are so-called "modified polyisocyanates", which contain carbodiimide groups, urethane groups, allophanate groups, isocyanurate groups, urea groups or biuret groups.

Further starting components are compounds having 2 or more isocyanate-reactive hydrogen atoms. They more particularly comprise compounds having a molecular weight of 400 to 10 000, and preferably from 2 to 8 hydroxyl groups with or without amino groups, thiol groups or carboxyl groups.

Optionally, further auxiliary and adjunct agents may also be used. For example, there may additionally be employed chemical blowing agents such as water and/or other volatile organic substances as physical blowing agent. Catalysts such as, for example, tertiary amines, such as dimethylcyclohexylamine, and/or organometallic compounds are also employable. It is possible to employ surface-active additives such as emulsifiers or foam stabilizers, for example siloxane-polyether copolymers, reaction retarders, cell regulators such as paraffins, fatty alcohols or dimethylpolysiloxanes, pigments, dyes, flameproofing agents such as phosphate esters or phosphonate esters, e.g. trischloroisopropyl phosphate. It is further possible to use stabilizers against ageing and weathering effects, plasticizers, fillers, dyes, antistats, nucleating agents, cell size regulator substances or biocidal actives.

Highly suitable catalysts are recited by way of example in the international patent application WO 96/14354. They include organic amines, aminoalcohols and aminoethers such as morpholine compounds, for example dimethylcyclohexylamine, diethanolamine, 2-dimethylaminoethyl 3-dimethylaminopropyl ether, 2-dimethylaminoethyl ether, 2,2-dimorpholinodiethyl ether, N,N-dimethylaminoethylmorpholine, N-dimethylmorpholine. Organometallic compounds such as, for example, tin, cobalt or iron compounds are also useful as catalyst. Tin dioctoate, cobalt naphthenate, dibutyltin dilaurate and iron acetonyl-acetate may be used for example.

The blowing agents may contain auxiliary and additive materials such as water, one or more catalysts, flameproofing agents, emulsifiers, foam stabilizers, binders, crosslinking agents, UV stabilizers, nucleating agents and optionally further propellant gases. The blowing agent may, for example, be added to the propolymers formed from polyol and poly- or diisocyanate, which is then foamed.

An initial advantage of the method is that the blowing agent composition applied has favourable properties with regard to ODP, GWP and photosmog. Compared with rigid polyurethane foams produced using purely hydrocarbons as blowing agents, the foams obtained in the method have a favourable thermal conductivity.

A particular advantage of rigid polyurethane foams obtainable by the method comes to bear at lower temperatures, usually at below about 15 DEG C. Astonishingly, the thermal conductivity of the rigid polyurethane foams obtainable by the method is not only better (i.e. lower) than that of foams produced from purely hydrocarbons, but even than that of foams produced with purely pentafluorobutane (HFC-365mfc). In substantially closed-cell rigid polyurethane foams which with blowing agent mixtures which include pentafluorobutane, preferably 1,1,1,3,3-pentafluorobutane, and at least 1 of the abovementioned further blowing agents, the blowing agent mixtures used appear to exert a synergistic effect in the direction of lower thermal conductivity, i.e. better thermal insulation. The rigid polyurethane foams obtainable by use of pentafluorobutane, preferably HFC-365mfc, and at least one more of the blowing agents indicated above are therefore particularly useful as insulation against cold in a temperature range below about 15 DEG C.

The production of thermoplastic foams will now be more particularly described.

The method can be used, for example, to foam the thermoplastics referred to in the abovementioned US patents 5,204,169 and 5,276,063 as being based on polymeric alkenylaromatics and the thermoplastics referred to in EP-A-0 436 847 as being based on polyphenylene ether compounds. Also foamable are thermoplastics based on polyethylene, polyvinyl chloride (PVC) and polyethylene terephthalate (PET) and polypropylene. The thermoplastics to be foamed in the method of the present invention are based with particular preference on polystyrene, polyethylene and polypropylene. It is very particularly preferable to employ polystyrene as thermoplastic polymer.

One method of preparing plastics based on polystyrene or polyethylene provides that when a) HFC-365mfc and b) 1,1,1,2-tetrafluoroethane (HFC-134a); 1,1,1,3,3-pentafluoropropane (HFC-245fa); 1,1,1,3,3,3-hexafluoropropane (HFC-236fa); or 1,1,1,2,3,3,3-heptafluoropropane (HFC-227ea), but no CO2 are present, the blowing agent composition contains or consists of less than 50 wt% of 1,1,1,3,3-pentafluorobutane and more than 50 wt% of 1,1,1,2-tetrafluoroethane; 1,1,1,3,3-pentafluoropropane; 1,1,1,3,3,3-hexafluoropropane or 1,1,1,2,3,3,3-heptafluoropropane. The same proviso also holds for this embodiment when no further blowing agent from the group of low-boiling, optionally halogenated hydrocarbons, chlorine and haloethers is present. Regarding preferred blowing agents, the observations made above in respect of PU foams apply.

The blowing agent composition is advantageously employed in an amount of 1 to 30 wt%, based on the combined mixture of thermoplastic polymer to be foamed and blowing agent composition.

One blowing agent composition that can be employed in the method contains or consists of a) pentafluorobutane, preferably 1,1,1,3,3-pentafluorobutane (HFC-365mfc) and b) at least one further blowing agent selected from the group comprising low-boiling aliphatic hydrocarbons, ethers and haloethers; difluoromethane (HFC-32); difluoroethane, preferably 1,1-difluoroethane (HFC-152a); pentafluoropropane, preferably 1,1,1,3,3-pentafluoropropane (HFC-245fa); hexafluoropropane, preferably 1,1,2,3,3,3-hexafluoropropane (HFC-236ea) or 1,1,1,3,3,3-hexafluoropropane (HFC-236fa); and heptafluoropropane, preferably 1,1,1,2,3,3,3-heptafluoropropane (HFC-227ea). A preferred composition contains or consists of: a) 1,1,1,3,3-pentafluorobutane (HFC-365mfc) and b) at least one further blowing agent selected from the group comprising ethane, propane, butane, pentane; difluoromethane (HFC-32); difluoroethane (HFC-152a); 1,1,1,3,3-pentafluoropropane (HFC-245fa); 1,1,1,3,3,3-

hexafluoropropane (HFC-236fa); and 1,1,1,2,3,3,3-heptafluoropropane (HFC-227ea). It preferably contains from 5 to 95 wt%, in particular from 10 to 70 wt% of 1,1,1,3,3-pentafluorobutane (HFC-365mfc).

Very highly suitable compositions contain or consist of: HFC-365mfc and HFC-152a; HFC-365mfc and HFC-32; HFC-365mfc, HF-152a and CO2; HFC-365mfc, HFC-32 and CO2; HFC-365mfc, HFC-152a and butane; HFC-365mfc, HFC-32 and butane; HFC-365mfc, HFC-32 and HFC-134a; HFC-365mfc and dimethyl ether; HFC-365mfc and pentane; HFC-365mfc and propane; HFC-365mfc and ethane; HFC-365mfc, pentane and CO2; HFC-365mfc, butane and CO2; HFC-365mfc, propane and CO2; HFC-365mfc, ethane and CO2.

The blowing agent composition contains with preference 1,1,1,3,3-pentafluorobutane (HFC-365mfc) and difluoromethane and/or 1,1-difluoroethane (HFC-152a), or it consists of these compounds. Particularly from 10 to 70 wt% of 1,1,1,3,3-pentafluorobutane and 90 to 30 wt% of 1,1-difluoroethane or difluoromethane are present, or it consists of these components in the amount ranges stated.

The blowing agent composition may additionally further contain from 2 to 50 wt% of liquefied carbon dioxide.

Also suitable is a blowing agent composition wherein when a) HFC-365mfc and b) 1,1,1,2-tetrafluoroethane (HFC-134a); 1,1,1,3,3-pentafluoropropane (HFC-245fa); 1,1,1,3,3,3-hexafluoropropane (HFC-236fa); or 1,1,1,2,3,3,3-heptafluoropropane (HFC-227ea), but no CO2 are present, the blowing agent composition contains or consists of less than 50 wt% of 1,1,1,3,3-pentafluorobutane and more than 50 wt% of 1,1,1,3,3-pentafluoropropane; 1,1,1,3,3,3-hexafluoropropane or 1,1,1,2,3,3,3-heptafluoropropane. The same proviso also holds when no further blowing agent from the group of low-boiling aliphatic hydrocarbons, ethers and haloethers is present.

Essentially closed-cell foamed polymers characterized by the presence of the disclosed blowing agent composition in the cells are likewise disclosed. They comprise in particular essentially closed-cell foamed thermoplastic polymers, preferably based on polystyrene, polyethylene, polypropylene, PVC or PET, in particular polystyrene.

The thermoplastic foams obtainable with the method have the advantage that they, compared with the use of, for example, HFC-134a as blowing agent, have an improved closed-cell content. In the case of polystyrene, improved processability of the polystyrene melt is observed as compared with the sole use of HFC-134a. The blowing agents exhibit adequate solubility. The blowing agents have no ODP and a low GWP. The effect on the formation of photosmog is extremely small.

One particular advantage of the rigid foams disclosed is that of improved properties in respect of thermal conductivity. The cells of the foam contain an increased level of residual blowing agent as compared with the sole use of HFC-134a, HFC-152a and HFC-32 as blowing agent.

The examples which follow are intended to illustrate the invention without limiting its scope.

Example 1:

Production of PU foams

To produce the PUR foam, one starting component used was a polyol mixture consisting of 40 parts by weight of an ethylenediamine/propylene oxide polyether (OH number 480), 60 parts by weight of a sorbitol/glycerol/propylene oxide polyether (OH number 490), 1 part by weight of foam stabilizer (DC 193 from Dow Corning Corp.) and 1.5 parts by weight of dimethylcyclohexylamine. Diphenylmethane diisocyanate was used as isocyanate component in a stoichiometric amount plus 10 wt%.

The PUR foams were produced on a low-pressure rig having an output performance of about 8 kg/min and allowing 3 component metering. A static mixer was used as mixing assembly.

a) Use of HFC-365mfc/152a

A blowing agent composition was used in an amount of 30 parts by weight, based on the polyol component. The blowing agent composition consisted of 70 parts by weight of HFC-365mfc and 30 parts by weight of HFC-152a. In addition, 1 part by weight of water was co-used as chemical blowing agent. The blowing agent composition produced a rigid PUR foam having a fine-cell structure and a density of about 32 kg/m^3 with minimal shrinkage.

b) Use of HFC-365mfc/32

A blowing agent composition was used in an amount of 30 parts by weight, based on the polyol component. The blowing agent composition consisted of 80 parts by weight of HFC-365mfc and 20 parts by weight of HFC-32. In addition, 1 part by weight of water was co-used as chemical blowing agent. The blowing agent composition produced a rigid PUR foam having a fine-cell structure and a density of about 28 kg/m^3 with minimal shrinkage.

c) Use of HFC-365mfc/152a/CO2

A blowing agent composition was used in an amount of 22 parts by weight, based on the polyol component. The blowing agent composition consisted of 70 parts by weight of HFC-365mfc and 30 parts by weight of HFC-152a. In addition to the blowing agent composition, 8 parts by weight of liquefied carbon dioxide were co-used as described in DE 44 39 082. Further, 1 part by weight of water was co-used as chemical blowing agent.

The blowing agent composition produced a rigid PUR foam having a fine-cell structure and a density of about 26 kg/m^3 with minimal shrinkage.

Example 2:

Producing a polystyrene foam

a) Use of HFC-365mfc/152a

200 kg of polystyrene (melt index 3.0 - 110) were mixed with 2 kg of talcum as nucleating agent and this mixture was metered into a customary extruder rig and melted. The polystyrene melt in the melt zone of the extruder was admixed with about 8 wt% of a blowing agent, based on the polystyrene, injected through a nozzle. The blowing agent mixture contained 30 wt% of HFC-365mfc and 70 wt% of HFC-152a.

In the mix zone, the polystyrene melt was mixed with the blowing agent composition until homogeneous and subsequently the mixture obtained was extruded through a die to obtain a closed-cell foam of uniform, fine-cell structure.

Both expanded polystyrene film and expanded polystyrene sheet were produced. One expanded polystyrene film obtained had a density of 38 kg/m^3, while one expanded polystyrene sheet obtained had a density of 35 kg/m^3.

b) Use of HFC-365mfc/32

Example 2a) was repeated except that the polystyrene melt was admixed with about 6 wt%, based on polystyrene, of the injected blowing agent. The blowing agent composition contained 30 wt% of HFC-365mfc and 70 wt% of HFC-32. One polystyrene film obtained had a density of 42 kg/m^3, while one expanded polystyrene sheet obtained had a density of 39 kg/m^3.

c) Use of HFC-365mfc/134a/152a

Example 2a) was repeated except that the polystyrene melt was admixed with about 8.5 wt%, based on polystyrene, of the injected blowing agent. The blowing agent composition contained 30 parts by weight of HFC-365mfc, 14 parts by weight of HFC-134a and 56 parts by weight of HFC-152a.

One polystyrene film obtained had a density of 40 kg/m^3, while one expanded polystyrene sheet obtained had a density of 38 kg/m^3.

d) Use of HFC-365mfc/152a/CO2

Example 2a) was repeated except that the polystyrene melt was admixed with about 5.5 wt%, based on polystyrene, of the injected blowing agent. The blowing agent composition contained 30 parts by weight of HFC-365mfc and 70 parts by weight of HFC-152a. In addition to the blowing agent composition, 8 parts by weight of liquefied carbon dioxide were co-used as described in DE 44 39 082.

One polystyrene film obtained had a density of 36 kg/m^3, while one expanded polystyrene sheet obtained had a density of 33 kg/m^3.

Example 3:

Producing a polyethylene foam

a) Use of HFC-365mfc/152a

200 kg of polyethylene (melt index 3.5 - 150) were extruded under conditions similar to those described in Example 2 for polystyrene. About 9 parts by weight of a blowing agent mixture, based on polyethylene, were injected. A blowing agent composition of 30 parts by weight of HFC-365mfc and 70 parts by weight of HFC-152a was used to obtain a fine-cell polyethylene foam with minimal shrinkage. The expanded polyethylene tube obtained had a density of 38 kg/m^3.

Patentkrav

1. Anvendelse af en drivmiddelsammensætning bestående af 10 til < 50 vægt-% 1,1,1,3,3-pentafluorbutan (HFC 365mfc) og > 50 vægt-% 1,1,1,3,3-pentafluorpropan (HFC 245fa) til fremstilling af polyurethanskum.