

Process for the filtration of substrate materials

The invention provides a process for the filtration of substrate materials that are suitable for the production of transparent injection moulded items, in particular for producing injection moulded items that are suitable for the production of optical data carriers, in particular those with high storage density, as well as other moulded parts of high optical quality. This process is suitable for both polycarbonates that are prepared using the phase interface method and also for polycarbonates that are produced using the melt transesterification process.

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The transparent injection moulded items may be e.g. transparent sheets, lenses, optical storage media, or else articles for the automotive glazing field such as e.g. light-scattering windscreens, for which high optical quality is required. Furthermore, this process is suitable for substrate materials from which extruded films in which a low-defect surface is important are produced.

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Transparent injection moulded items are of importance in particular in the glazing and storage media fields.

20 Optical data recording materials have recently been used to an increasing extent as a variable recording and/or archiving medium for large volumes of data. Examples of this type of optical data memory are e.g. CD, super-audio-CD, CD-R, CD-RW, DVD, DVD-R, DVD+R, DVD-RW, DVD+RW and BD.

25 Transparent thermoplastic materials, such as for example polycarbonate and chemical modifications thereof, are typically used for optical storage media. A substrate material that is suitable in particular for write-once-read-many times and also for write-many times optical discs is primarily aromatic polycarbonate, in particular polycarbonate based on bisphenol A. Polycarbonate is also suitable for the production of moulded parts for the automotive glazing field, such as e.g. for light-scattering windscreens. This thermoplastic material has exceptional mechanical stability, is less susceptible to changes in dimensions and is characterised by high transparency and impact resistance.

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In order to memory larger volumes of data on a data carrier, formats such as the DVD were developed after CDs. These enable for example the storage of feature films as a result of the increased storage capacity. Thus, a DVD has 6-8 times the
5 take-up capacity of a CD. This was made possible by the use of smaller pits, smaller track spacings and a reduction in the laser wavelength from about 780 nm to about 655 nm for DVDs. Furthermore the numerical aperture of the optical readout system was increased.

10 The development of new formats such as e.g. HD-DVD (high density DVD) or the so-called blu-ray disc (BD) again operate with shorter laser wavelengths. In the case of a BD, readout takes place through a transparent cover layer material (e.g. PC film).

15 This means that the requirements placed on the substrate through which the radiation passes or the cover material through which the radiation passes are increasingly stringent. The higher the storage density of the particular medium, the higher are the requirements (e.g. lower particle contents) placed on the quality of the substrate material. Defects in the substrate layer through which radiation is transmitted and in
20 the cover material through which radiation is transmitted lead to errors in the readout process.

Thus, defects, in particular those that can interact with the laser beam of the readout system, are of particular relevance with regard to an error-free readout process.
25 These include, as is well-known, foreign particles such as e.g. dust particles or metal particles that can absorb and/or scatter the laser beam. As a result of lowering the wavelength of the readout laser, those particles whose absorption or fluorescence lies within the wavelengths of the laser used, can also cause problems. In the case of a HD-DVD (high density DVD) or blu-ray disc, for example, these are wavelengths
30 between 400 nm and 410 nm.

High-quality extruded films for which these substrate materials are also suitable are used primarily in the electronics sector, for decorative and functional blinds in the

domestic sector and as covering films, e.g. for sports items, for ID cards and for blister packs. Other areas of application are in the field of automobile construction, e.g. as bodywork parts or external mirrors, or in the field of telecommunication such as e.g. mobile phone cases and mobile phone keyboards. The films are characterised by high transparency, impact resistance and thermal stability. Another critical quality feature is the surface quality of the film. In particular in the case of high-quality housing and display covers, imperfections in the surface that lead to increased rates of rejection are very noticeable.

The melt filtration of polymers is known in principle and is described, for example, in EP-A 0806281 and EP-A 0220324. The melt filtration of polycarbonate in particular is also described, for example, in EP-A 1199325. The filtration of highly viscous polymer melts is quite a critical process because high pressures are often used and the shear forces in the filter can lead to shear heating that can then result in thermal decomposition of the polycarbonate. Depending on the residence time in the filter and the temperature of the polymer melt, interaction with the large metal surface can also result in damage to the polycarbonate. Depending on the pressure and throughput, there is a risk that fluorescent particles are forced through the filter medium or are generated due to the factors mentioned above.

The object of the invention was therefore to provide a process for the filtration of polymer melts, in particular polycarbonate melts, that enables the separation of fluorescent particles from polycarbonate and enables a substrate material with a low fluorescent particle content to be obtained.

JP 04342760 (1992) describes a melt filtration process for highly viscous polycarbonate. The melt filtration process described therein is performed using filter media with a pore size of 20 μm . The filter medium is not specified in any more detail. The retention of fluorescent particles is not mentioned.

EP-A 1156071 describes a passivation process for filter media with weak acids such as e.g. phenol. This achieves an improvement in the yellowness index of the filtered material. Fluorescent particles are not described. This method and a filtration process

using these types of pretreated filters is not suitable for a reduction in the number of fluorescent particles.

5 JP 20011310935 describes a melt filtration process for the removal of impurities in polycarbonate. The separation of fluorescent particles is not described. The temperature of the melt that passes through the filter is 280 to 340°C. Melt filtration is performed using filters with a pore size of 0.5 to 50 µm. The filter medium is not specified precisely. Thus, for example, the thickness of the filter medium is not described. However, the particle content also depends on the concentration of free
10 bisphenol. In contrast to that, in the process according to the invention described here the bisphenol content is of lesser importance. Pretreatment of the filter media is not described.

15 EP-A 1199325 describes a melt filtration process. Melt filtration is performed through filter media with a pore size ≤ 40 µm. However, nothing is said with regard to the thickness of the filter layer, which is a critical quantity in order for the particles described here to be retained. No method is reported that retains fluorescent particles with specific viscous properties. The filter media are not pretreated.

20 JP 2003048975 describes the production of polycarbonate in a melt transesterification process. In this process the molten raw materials are subjected to filtration through a passivated filter. There is no mention of the formation and separation of fluorescent particles. The method according to the invention differs
25 from that described in JP2003048975 because in the Japanese laid-open document the raw materials and not the polymer melts are filtered.

30 None of the documents mentioned above describes the filtration process according to the invention or mentions the concentration of fluorescent particles in the polycarbonate. The methods mentioned above do not lead to an effective reduction in the number of fluorescent particles in polycarbonate.

It is known that substrate materials for optical data storage contain foreign bodies. EP-A 379130 states for example that these foreign bodies can have a negative effect on writing to or reading from an optical data carrier using a laser beam. A substrate material has a good quality when a certain particle content (particle index), as
5 described in this patent, lies below a certain value. The foreign bodies may be dust or charred material or metal abrasion. These foreign bodies, which are clearly differentiated from the substrate material, are detected in a solvent in which the substrate material itself is soluble. These foreign bodies that can be detected in this solvent are not the subject-matter of the present invention. The foreign particles
10 described here cannot be detected in a solvent in which the substrate material is soluble because they have a similar refractive index to that of the polycarbonate solution being measured. Thus, these particles are not detected for example by the Hiac Royco test in accordance with the prior art, as described e.g. in DE 102 48 951.

15 JP-A-020135222 describes foreign bodies with a gel-like character that have a refractive index different from that of the matrix. These particles can also reduce the quality of the optical data memory. These particles can be greatly reduced in an extrusion process, for example by adding water. These particles differ from the ones
20 described here because the refractive index of the particles described here does not differ substantially from that of the matrix. Furthermore, the particles described here cannot be removed, nor their concentration lowered, in an extrusion process by the addition of water.

25 It was found that substrate materials, in particular substrate materials based on polycarbonate, contain particles that exhibit fluorescence within the critical absorption spectrum described above for a blue laser (400 to 410 nm). Surprisingly, it was found that a large proportion of these fluorescent particles have different rheological properties from those of the polymer matrix (polycarbonate) itself. These defects found in substrate materials can lead to flow distortions in the final injection
30 moulded part, such as for example an optical disc. Thus, the rheological properties of these particles differs from that of the polymer matrix.

The fluorescent particles in granules and the fluorescent flow distortions in injection moulded items are characterised by the following properties:

5 Measurements of the mechanical properties of the particles produce a higher modulus and a greater hardness than those of the matrix material (polycarbonate). The hardness of the particles is up to about 0.3 GPa higher than that of the matrix. The values for hardness were determined using a nanoindenter made by Hysitron on a disc surface and over one cross-section of the disc (particles vs. matrix). Depending on the position of the fluorescent particles in the injection moulded item,
10 flow distortions and thus defects in the surface of the injection moulded item are initiated. This applies in particular below certain cycle times, mainly below 3.5 and in particular below 3 seconds. These flow distortions, which fluoresce of themselves, are relevant not only for optical data memories through which a blue laser beam passes, but also for other injection moulded items such as e.g. lenses or light-
15 scattering windscreens, because these flow distortions generally reduce the optical quality of the surface. In the case of optical data memories, these flow distortions are detected by a scanner during the production process. The faulty discs are rejected.

The optical defect on an injection moulded item can in the case of a disc attain a
20 length of several millimetres. The defect is oriented in the direction of flow, i.e. in the case of optical discs for optical data carriers it is oriented radially. There is a fluorescent flow distortion within this optical defect. It was found that the fluorescent flow distortions, e.g. on discs, have a length/width ratio of 2 – 30, on average of 5 – 15. The length of the fluorescent flow distortions is between 10 and
25 200 μm . These values depend strongly on the machine parameters set during the injection moulding process, in particular on the cycle time.

It was also found that the fluorescent bodies exhibit relaxation behaviour. After these fluorescent flow distortions have been conditioned for 2 minutes at 300°C, the
30 length/width ratio is found to be about 1, i.e. the longitudinal fluorescent flow distortions relax to give a spherical body; i.e. these fluorescent bodies can be thermoplastically deformed, but do not have sufficient solubility in the polymer matrix.

The fluorescent flow distortions exhibit a certain swelling behaviour. If these flow distortions are treated with dichloromethane for 5 minutes, then the area of the fluorescent region increases. The increase in volume of the defect was determined therefrom – giving an increase by a factor of 1.1 to 3.5 (the spherical volume of the defect was determined from the radius of the circle of equivalent area). On average, an increase by a factor of about 2 is obtained. The polymer matrix itself dissolves completely in dichloromethane in the case of polycarbonate, whereas the fluorescent body remains undissolved.

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Furthermore it was found that the fluorescent particles take on certain colour indices. For this purpose the particles were irradiated with light from a mercury vapour lamp through a step filter with a passage from 470 nm upwards. The colour of the particles was determined using a digital colour camera, a Zeiss Axiocam HRc, incorporated in a Zeiss Axioplan 2 microscope, using the HSI (hue, saturation, intensity) colour model. The method is described in e.g. "Digitale Bildverarbeitung mit dem PC", Hans-Jürgen Schlicht, Addison-Wesley, 1993. If the colour of the fluorescing particles is measured at a given setting for the illumination, then the following values are found: the hue value is on average about 80°, the colour saturation is on average 150 digits and the colour intensity is on average 190 digits. The colour of the matrix in the case of polycarbonate (compact disc) is given by a hue value of about 75°, a colour saturation of on average 133 digits and a colour intensity of on average 36 digits. In order to give the correct colour site of the particles and the matrix, the values for hue, saturation and intensity have to be combined. In this way only particles with a characteristic colour are counted, i.e. other particles, such as e.g. dust, are not taken into account.

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The larger the number of fluorescent particles of a certain size in the polymer granules, the greater is the probability of obtaining flow distortions in the final injection moulded item. This raises the rejection rate of the particular product. Surprisingly, it was found that not all fluorescent particles lead to flow distortions, but only particles of a certain size. Fluorescent particles that lay below the flow

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distortions were prepared from optical discs and relaxed in the melt as described above. Only spherical particles with a diameter of $\geq 10 \mu\text{m}$ were determined.

5 Starting from the prior art, the object therefore existed of separating out the problematic particles described above and thereby obtaining an ultrapure substrate material, preferably made of polycarbonate, that is suitable for the production of data carriers, in particular those data carriers that are read with blue laser light, as well as the production of high-quality optical injection moulded items and high-quality extrusion moulding compounds.

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The object is achieved by providing a special melt filtration process for separating out the fluorescent particles. This is particularly surprising because particles with the viscous properties described above can be separated out only with great difficulty because they can be deformed as a result of their properties. Using a special deep-
15 filtration process it was possible to produce a substrate material that is suitable for the production of data carriers, in particular those data carriers that are read with blue laser light, as well as for the production of high-quality injection moulded items.

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The invention provides a process for the filtration of polycarbonate, characterised in that the melt of polycarbonate with an average molecular weight (weight average) of 14,000 – 30,000 g/mole, preferably 15,000 – 29,000g/mole and particularly preferably 16,000 – 22,000 g/mole (determined by gel permeation chromatography) is filtered through at least one metal non-woven structure consisting of more than one
25 layer (stratum) of thin metal wires. The wires are stapled to form layers and may optionally be stabilised by thicker supporting wires. The metal non-woven structure consists of wires of e.g. V4A steel, e.g. with the material number 1.4571. The supporting wires may consist of e.g. V4A 1.4571 or V2A 1.4541 or steel with the material number 1.4310. The non-woven structure has a thickness of 5 mm to 0.1
30 mm, preferably 1 mm to 0.2 mm. The non-woven structure is built up from alternating layers of different density (i.e. with different pore sizes). The densest layer is preferably located in the middle of the non-woven structure. The densest

layer is preferably 0.05 to 0.2 mm thick. Furthermore, the densest layer preferably consists of thin wires with a diameter of 1 – 20 μm , more preferably 2 – 10 μm , particularly preferably 2 – 7 μm . The less dense layer is preferably 0.05 to 0.3 mm thick. The wires in this layer have a diameter of 1 – 20 μm , preferably 2 – 10 μm , particularly preferably 2 – 7 μm . The pore size in the less dense layers is preferably 20 – 60 μm , that in the densest layer preferably 1 – 10 μm . These wires may be smooth or have a surface structure that can be achieved by special methods of treatment such as by treating with acid, e.g. nitric acid or citric acid, or by electropolishing. There are preferably 2 to 10, particularly preferably 2 to 8 and in particular 2 to 6 layers. Other support gratings, perforated sheets, expanded metal or similar materials that are used to support the non-woven structure can be attached to the front and rear faces of the metal non-woven structure.

The filtration process is generally performed under pressure, preferably at pressures of 5 to 35 bar, in particular at 10 to 30 bar.

The filter non-woven structure and all metal surfaces of the filter may be thermally pretreated. Thermal pretreatment may take place in a protective gas atmosphere such as nitrogen or argon or in an oxidative atmosphere such as air, preferably in an oxidative atmosphere.

Thermal pretreatment is generally performed at temperatures of 200 to 1200°C, depending on the type of conditioning and the corresponding conditioning temperature. The duration of thermal pretreatment is generally 1 minute to 48 hours, preferably 2 minutes to 30 hours and in particular 10 minutes to 24 hours, and depends on the type of conditioning.

Thermal pretreatment may be carried out by annealing the metal surface in a flame, by conditioning in a muffle furnace or in a fluidised bed or by conditioning in a circulating air oven.

When annealing the metal surface in a gas flame, the temperature is generally between 600 and 1500°C, preferably between 800 and 1200°C, when conditioning in

a muffle furnace the temperature is between 300 and 1000°C, preferably between 600 and 900°C, and when conditioning in a circulating air oven the temperature is 200 to 500°C, preferably 300 to 400°C.

5 The duration of thermal pretreatment at temperatures above 600°C is generally 1 minute to 1 hour, in the temperature range 300 to 700°C it is generally 30 minutes to 48 hours, preferably 30 minutes to 30 hours, in particular 2 to 25 hours, and at temperatures of 300 to 500°C the duration is generally 1 to 48 hours, preferably 2 to 30 hours, in particular 4 to 25 hours. Flaming the metal parts treated by annealing
10 may take place in a sand or fluidised bed.

The metal surfaces of the filter non-woven structure are particularly preferably conditioned at 200 to 500°C, preferably 250 to 450°C, in particular 300 to 400°C, for up to 35 hours, preferably 4 to 30 hours, in particular 8 to 26 hours. A temperature of
15 350°C for 24 hours is very particularly preferred.

Using this method of filtration, the number of fluorescent particles with a particle diameter of 5 – 250 µm in the polycarbonate can be greatly reduced. In particular, the number of particles that have a particle diameter greater than 50 µm is greatly
20 reduced.

The polycarbonates according to the invention are produced, inter alia, using the phase interface process. Many variants of this process for synthesising polycarbonate have been described in the literature; for example reference may be made to H.
25 Schnell, Chemistry and Physics of Polycarbonates, Polymer Reviews, vol. 9, Interscience Publishers, New York 1964, p. 33 et seq., to Polymer Reviews, vol. 10, "Condensation Polymers by Interfacial and Solution Methods", Paul W. Morgan Interscience Publishers, New York 1965, chap VIII, p. 325, to Drs. U. Grigo, K. Kirchner and P.R. Müller "Polycarbonate" in Becker/Braun, Kunststoff-Handbuch,
30 vol. 3/1, Polycarbonate, Polyacetale, Polyester, Celluloseester, Carl Hanser Verlag, Munich, Vienna 1992, pp. 118-145 as well as to EP-A 0 517 044.

In accordance with this process the phosgenation of a disodium salt of a bisphenol

(or a mixture of different bisphenols) added to an aqueous/alkaline solution (or suspension) is carried out in the presence of an inert organic solvent or solvent mixture that forms a second phase. The oligocarbonates that are formed and which are present mainly in the organic phase are condensed with the aid of suitable catalysts, to give high molecular weight polycarbonates dissolved in the organic phase. The organic phase is finally separated and the polycarbonate is isolated therefrom by a variety of working-up steps.

Diphenols that are suitable for the production of polycarbonates to be used in accordance with the invention are, for example, hydroquinone, resorcinol, dihydroxydiphenyl, bis-(hydroxyphenyl)-alkanes, bis-(hydroxyphenyl)-cycloalkanes, bis-(hydroxyphenyl)-sulfides, bis-(hydroxyphenyl)-ethers, bis-(hydroxyphenyl)-ketones, bis-(hydroxyphenyl)-sulfones, bis-(hydroxyphenyl)-sulfoxides, α,α' -bis-(hydroxyphenyl)-diisopropylbenzenes, and their alkylated, ring-alkylated and ring-halogenated compounds.

Preferred diphenols are 4,4'-dihydroxydiphenyl, 2,2-bis-(4-hydroxyphenyl)-1-phenylpropane, 1,1-bis-(4-hydroxyphenyl)-phenylethane, 2,2-bis-(4-hydroxyphenyl)-propane, 2,4-bis-(4-hydroxyphenyl)-2-methylbutane, 1,3-bis-[2-(4-hydroxyphenyl)-2-propyl]-benzene, (bisphenol M), 2,2-bis-(3-methyl-4-hydroxyphenyl)-propane, bis-(3,5-dimethyl-4-hydroxyphenyl)-methane, 2,2-bis-(3,5-dimethyl-4-hydroxyphenyl)-propane, bis-(3,5-dimethyl-4-hydroxyphenyl)-sulfone, 2,4-bis-(3,5-dimethyl-4-hydroxyphenyl)-2-methylbutane, 1,3-bis-[2-(3,5-dimethyl-4-hydroxyphenyl)-2-propyl]-benzene, 1,1-bis-(4-hydroxyphenyl)-cyclohexane and 1,1-bis-(4-hydroxyphenyl)-3,3,5-trimethyl-cyclohexane (bisphenol TMC) and mixtures of these.

Particularly preferred diphenols are 4,4'-dihydroxydiphenyl, 1,1-bis-(4-hydroxyphenyl)-phenylethane, 2,2-bis-(4-hydroxyphenyl)-propane, 2,2-bis-(3,5-dimethyl-4-hydroxyphenyl)-propane, 1,1-bis-(4-hydroxyphenyl)-cyclohexane and 1,1-bis-(4-hydroxyphenyl)-3,3,5-trimethylcyclohexane (bisphenol TMC) and mixtures of these.

These and other suitable diphenols are described in e.g. US-A –PS 2 999 835, 3 148 172, 2 991 273, 3 271 367, 4 982 014 and 2 999 846, in German laid-open specifications 1 570 703, 2 063 050, 2 036 052, 2 211 956 and 3 832 396, French patent 1 561 518, in the monograph "H. Schnell, Chemistry and Physics of Polycarbonates, Interscience Publishers New York 1964, p. 28 et seq.; p. 102 et seq.", and in "D.G. Legrand, J.T. Bendler, Handbook of Polycarbonate Science and Technology, Marcel Dekker, New York 2000, p. 72 et seq."

In the case of homopolycarbonates only one diphenol is used, whereas in the case of copolycarbonates several diphenols are used, wherein obviously the bisphenols used, as also all other chemicals and auxiliary substances added for the synthesis, may be contaminated with impurities arising from their own synthesis, handling and storage procedures, although it is desirable to work with raw materials that are as clean as possible.

The monofunctional chain terminators required to control the molecular weight, such as phenol or alkyl phenols, in particular phenol, p-tert.butylphenol, iso-octylphenol, cumylphenol, their chlorinated esters or acid chlorides of monocarboxylic acids or mixtures of these chain terminators, are either added with the bisphenolate(s) in the reaction or else at any other point in the synthesis, as long as phosgene or chlorinated carboxylic acid end groups are still present in the reaction mixture or, in the case where acid chlorides and chlorinated esters are used as chain terminators, as long as sufficient phenolic terminal groups of the polymers are available. However, the chain terminator(s) is/are preferably added after phosgenation at a site or a time when phosgene is no longer present but the catalyst has still not been metered in, or they are metered in before the catalyst, together with the catalyst or in parallel with the catalyst.

Any branching agents or branching agent mixtures being used are added to the synthesis in the same way, but generally before the chain terminators. Trisphenols, quaternary phenols or acid chlorides of tri- or tetracarboxylic acids are generally used, or else mixtures of polyphenols or acid chlorides.

Some of the compounds with three or more than three phenolic hydroxyl groups that can be used are for example

phloroglucine,

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4,6-dimethyl-2,4,6-tri-(4-hydroxyphenyl)-heptene-2,

4,6-dimethyl-2,4,6-tri-(4-hydroxyphenyl)-heptane,

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1,3,5-tri-(4-hydroxyphenyl)-benzene,

1,1,1-tri-(4-hydroxyphenyl)-ethane,

tri-(4-hydroxyphenyl)-phenylmethane,

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2,2-bis-[4,4-bis-(4-hydroxyphenyl)-cyclohexyl]-propane

2,4-bis-(4-hydroxyphenyl-isopropyl)-phenol

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tetra-(4-hydroxyphenyl)-methane.

Some other trifunctional compounds are 2,4-dihydroxybenzoic acid, trimesic acid, cyanuric chloride and 3,3-bis-(3-methyl-4-hydroxyphenyl)-2-oxo-2,3-dihydroindole.

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Preferred branching agents are 3,3-bis-(3-methyl-4-hydroxyphenyl)-2-oxo-2,3-dihydroindole and 1,1,1-tri-(4-hydroxyphenyl)-ethane.

The catalysts used in the phase interface process are tertiary amines, in particular triethylamine, tributylamine, trioctylamine, N-ethylpiperidine, N-methylpiperidine, N-i/n-propylpiperidine; quaternary ammonium salts such as tetrabutylammonium / tributylbenzylammonium / tetraethylammonium hydroxide / chloride / bromide / hydrogen sulfate / tetrafluoroborate; as well as the phosphonium compounds corresponding to these ammonium compounds. These compounds are described in

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the literature as typical phase interface catalysts, are commercially available and are familiar to a person skilled in the art. The catalysts may be added to the synthesis individually, as a mixture or else alongside each other or in sequence, optionally also before phosgenation, but preferably are metered in after the introduction of phosgene, unless an onium compound or mixture of onium compounds is used as catalyst, when addition prior to the introduction of phosgene is preferred. The catalyst(s) may be metered in in bulk, in an inert solvent, preferably the one used for the polycarbonate synthesis, or else as an aqueous solution, and in the case of tertiary amines this then takes place in the form of their ammonium salts with acids, preferably inorganic acids, in particular hydrochloric acid. When using several catalysts or when portions of the total amount of catalyst are metered in, naturally different modes of metering may take place at different sites or at different times. The total amount of catalysts used is between 0.001 and 10 mole.% with respect to the moles of bisphenols used, preferably 0.01 to 8 mole.%, particularly preferably 0.05 to 5 mole.%.

In addition, the production of polycarbonates from diaryl carbonates and diphenols using the well-known polycarbonate process in the melt, so-called melt transesterification, is also possible, as described e.g. in WO-A 01/05866 and WO-A 01/05867. In addition transesterification processes (acetate process and phenyl ester process) are described for example in US-A 34 94 885, 43 86 186, 46 61 580, 46 80 371 and 46 80 372, in EP-A 26 120, 26 121, 26 684, 28 030, 39 845, 91 602, 97 970, 79 075, 14 68 87, 15 61 03, 23 49 13 and 24 03 01 as well as in DE-A 14 95 626 and 22 32 977.

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The following examples explain the invention but do not restrict it.

Examples

Method for the determination of the fluorescent particle content in polycarbonate:

- 5 Analysis of the content of fluorescent particles is performed by filtration of the polycarbonate sample concerned (50 g), dissolved in dichloromethane (LiChrosolv; Merck: 1.06044 K33506244 430) (700 ml), through a Teflon filter membrane (Bohlender GmbH, D-97847, Grünsfeld) with a 5 µm pore diameter and a filter membrane thickness of 1 mm. The filter discs are dried under vacuum and protected
- 10 from surrounding dust by a covering. After filtration the filter surface is investigated (scanned) using an Axion 2 fluorescence microscope from Zeiss AG, Germany. An excitation wavelength of 400 – 440 nm, an illumination time of 40 ms per scan and 25- fold total magnification are used. The fluorescent particles are detected and the data is evaluated using image processing software (KS 300 3.0 from Zeiss AG).
- 15 Only particles with a characteristic colour are counted, i.e. other particles such as dust are not taken into account (determined by the HIS colour model, see above). The colour parameters for detecting fluorescent particles are set so that they are equivalent to the parameters of particles found in flow distortions in optical discs. The surface of the filter is scanned automatically using a computer-controlled object
- 20 lens table (Zeiss AG).

In this way, and under the conditions cited above (wavelength, total magnification, illumination time), a coherent fluorescing region on the Teflon filter is automatically detected and counted as 1 count. The individual fluorescent particles that are found

25 on the Teflon filter are counted. The total number of fluorescent particles is divided by the weight of the polycarbonate melt weighed out in the individual batch and this gives the number of particles (fluorescent particles) with respect to 1 gram of polycarbonate (counts/g).

- 30 An aromatic polycarbonate based on bisphenol A and tert-butylphenol as chain terminator with a melt volume rate MVR = 71 cm³/10 min (at 300°C; 1.2 kg) was used as the test material.

The polycarbonate granules, predried at 120°C in a vacuum drying cabinet, are melted at different temperatures in an extruder (Brabender 35/17D, Karg Industrietechnik, 82152 Krailling), with data processing/evaluation Plasti-Corder PL2000 version 2.6.9, and processed with different throughputs. The melt is filtered
5 on the one hand using the process according to the invention and on the other hand using a conventional filtration process (comparison).

Example 1 (comparison example – 0 sample – no filtration)

10 The polycarbonate mentioned above is melted in an extruder (see above). The heating system for the individual heating zones is set at 305°C (zone 1), 310°C (zone 2) and 315°C (zone 3). At the start, the extruder is purged for 3 minutes at a throughput of 6 kg/h. Then the throughput is reduced to 1.2 kg/h and the extruder is purged for 15 minutes at this throughput. The temperature of the polymer melt at the
15 die plate is about 295-305°C. A sample is taken from the polymer melt.

The number of fluorescent particles in the polycarbonate obtained in this way is determined using the method described above. The result gives 15.2 counts/g of fluorescent particles and thus a high value for the number of fluorescent particles.

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Example 2 (comparison example, with 25 µm filter)

The test in example 1 was performed, but a melt filter was used upstream of the die plate. For this purpose, a thin-layered stainless steel filter was used (thickness on
25 average 30 µm; wire diameter about 10 µm), in which the supporting gauze had been removed, i.e. only the filter membrane was used, a circular piece with a diameter of 41.2 mm being cut out. The pore size in the filter was 25 µm. In the flow direction of the polymer first this filter membrane, then a supporting gauze (expanded metal with a mesh size of 0.5 x 1.2 mm²), followed by a supporting ring with wire gauze with a
30 mesh size of 1.2 mm² and then a perforated plate with holes of 2.2 mm were installed. Finally the die plate was installed and fixed in place with a cap nut. The diameter of the active filtration area is 30 mm. Using this arrangement ensures that the filter is supported, i.e. is not damaged, and that covering is achieved.

The polymer granules are melted under the conditions described above. Melt samples are taken after 35 minutes and after 60 minutes (reckoned from the time of installation of the filter). The first sample, after 35 minutes, had a fluorescent particle content of 7.8 counts/g. The second sample after 60 minutes had a value of 8.3 counts/g. The pressure upstream of the filter was about 1 bar. This result demonstrates that the incorporation of the filter mentioned above reduces the number of fluorescent particles only slightly.

10 **Example 3** (comparison example)

The test in example 1 was performed, but a melt filter was inserted into the die plate. For this purpose, a stainless steel non-woven structure made from the material with material number 1.4571 was used. The non-woven structure consisted of two layers.

15 In the flow direction of the melt the sequence was firstly a less dense layer with a thickness of 0.15 mm. The pore size of the less dense layer was on average 30 μm . The more dense layer adjoined the less dense layer and had a thickness of 0.15 mm. The pore size of the more dense layer was on average about 10 μm . The metal fibres had a diameter of about 5 – 7 μm . The thickness of the filter was 0.3 mm in total.

20 The diameter of the non-woven filter structure was 28.4 mm. The non-woven filter structure is the first layer in the sequence in the flow direction of the polymer melt. Following this are installed a support gauze (expanded metal with a mesh size of 0.5 x 1.2 mm²), then a support ring with a wire gauze with a mesh size of 1.2 mm and then a perforated sheet with holes of 2.2 mm. Finally the die sheet is installed and

25 fixed in place with a cap nut. Using this arrangement ensures that the filter is supported, i.e. is not damaged, and covering is achieved. The diameter of the active filtration area is 19 mm. The polymer granules are melted under the conditions described above. After 25 minutes (reckoned from the time of insertion of the melt filter) a sample is taken from the polymer melt. The pressure upstream of the non-

30 woven filter structure is 55 - 60 bar.

The number of fluorescent particles in the polycarbonate obtained in this way is determined using the method described above.

Evaluation gave 8.7 counts/g.

5 This shows that under conditions that are outside the conditions in accordance with the invention, effective separation of the fluorescent particles is possible to only an unsatisfactory extent.

Example 4 (according to the invention, after insertion of the filter)

10 The test in example 1 was performed, but a melt filter was inserted into the die plate. The metal non-woven structure had the same structure as described in example 3. However, the diameter of the non-woven filter structure was 41.2 mm. The diameter of the active filtration area was 30 mm. The polymer granules are melted under the conditions described above. After 25 minutes (reckoned from the time of insertion of
15 the melt filter), the first sample is taken, and a second sample is taken after a further 25 minutes. The pressure upstream of the non-woven filter structure is 20 – 25 bar.

The number of fluorescent particles in the polycarbonate obtained in this way is determined using the method described above.

20

Evaluation of the first sample gave 1.56, that of the second sample 1.18 counts/g, of fluorescent particles.

25 This demonstrates that the method of melt filtration according to the invention leads to a clear reduction in the number of fluorescent particles in the polymer melt.

Example 5 (according to the invention, with a passivated filter)

30 The test in example 1 is performed, but the melt filter is first conditioned for 24 hours at 350°C in a circulating air oven. The filter is inserted in the way described in example 4. The polymer granules are melted under the conditions given above. A sample is taken after 3 hours.

Evaluation of the sample gave 0.47 counts/g of fluorescent particles.

The advantages of the filtration method according to the invention can be seen quite clearly from the significantly lowered number of fluorescent particles.

Claims

1. Process for the filtration of polycarbonate melts, characterised in that the melt of polycarbonate with an average molecular weight (weight average) of 14,000 to 30,000 g/mole is filtered through at least one non-woven metal structure with a thickness of 5 mm to 1 mm, consisting of two or more layers, wherein at least one layer is 0.05 to 0.2 mm thick and has a pore size of 1 to 10 μm .
5
2. Process according to Claim 1, wherein 2 to 10 layers are used.
10
3. Process according to Claim 1, wherein the thickness of the non-woven metal structure is 1 mm to 0.2 mm.
4. Process according to Claim 1, wherein the layer that is 0.05 to 0.2 mm thick consists of wires with a diameter of 1 – 20 μm .
15
5. Process according to Claim 1, wherein the less dense layer is 0.05 to 0.3 mm thick.
20
6. Process according to Claim 5, wherein the pore size of the less dense layer is 20 to 60 μm .
7. Process according to Claim 1, wherein filtration is performed at pressures of 5 to 35 bar and temperatures of 220 to 400°C.
25
8. Process according to Claim 1, wherein the filter gauze is made of stainless steel.
9. Process according to Claim 1, wherein the non-woven filter structure is thermally pretreated in an oxidative atmosphere.
30

10. Process according to Claim 9, wherein the non-woven filter structure is pretreated for 4 to 35 hours in an oxidative atmosphere at temperatures of 300 to 400°C.
11. Process according to Claim 1 for removing fluorescent particles in polycarbonate.