Installation and method for producing a fiber from a polymer solution

The invention relates to a plant and a process for producing hollow fibre membranes for fluid separation, in particular for gas separation.

The prior art discloses processes for production of membrane material for fluid separation, and such membrane materials based on polyimides are described in, for example, WO 2014/202324 A1 or WO 2011/009919. Such membrane materials which are integrally asymmetric membranes, so-called "integrally skinned asymmetric membranes" (ISA membranes), comprise a separatory layer of 10 to 200 nm. It is well known that polyimides represent a class of polymer materials which make it possible to achieve gas separation of a fluid mixture at a high permeance of one of the fluids and/or at high selectivity. Polyimides are also characterized by very good mechanical, chemical and thermal resistance in applications for gas separation.

It is known that polyimides and also other glass-like polymers which are used as membrane material for gas separation exhibit a decrease in the initial separation performance, for example by plasticization (softening) ("Quantification of sorption, diffusion, and plasticization properties of cellulose triacetate films under mixed-gas CO2/CH4 environment" Genduso et al.; J. of Membrane Science, volume 610, 01.09.2020, article number 118269), by physical ageing ("Physical aging of thin glassy polymer films monitored by gas permeability" Huang Y. et al.; Polymer, vol. 45, Issue 25, pages 8377 – 8393, November 2004) and/or by contact with impurities in the feed stream ("Properties of a polyimide gas separation membrane in natural gas streams"; White et al.; J. of Membrane Science, volume 103, issue 1-2, pages 73 – 82, 14.07.1995).

The aforementioned disadvantages can be partly or even completely avoided if the hollow fibre membrane is heated above 300°C, as described for example in WO 2011/009919 A1 and US 9,873,093 B2. It is further proposed therein, for establishment of defined, high permeances and selectivity, to provide a defined gradient during heating as part of the heat treatment, this gradient for example according to WO 2011/009919 A1 comprising three stages in a recirculating drying cabinet (convection oven), namely at 70°C (0.5 h), 150°C (2 h) and 250°C (12 h).

For further improvement, WO 2014/202324 A1 proposes drying hollow fibre preferably at a temperature in the region of 50 and 100°C, followed by heat treatment. The drying is said to remove for example water, isopropanol or hexane, with the result that the total water and/or residual solvent content is in the range from 0% to 5% by weight. This is because an excessively high residual content of water and solvent in the heat treatment of whole membrane bundles leads to the membranes sticking to one another. Excess water can also lead to hydrolysis and thus to chain scission and a mechanically unstable membrane. According to WO 2014/202324 A1 it is advantageous if the maximum content of water before commencement of heat treatment is below 5% by weight, irrespective of the fact that any water content would evaporate during the heat treatment itself.

It is proposed to provide a drying unit and a heat treatment unit, wherein the heat treatment has to be carried out at a temperature in the preferred range from 140°C to 180°C under vacuum in the range from 0.6 to 0.9 bar and under an inert gas atmosphere, wherein a very long heat treatment time of preferably 10 to 16 hours is required.

Disadvantages of these previously known and fundamentally proven process regimes and plants are that a relatively elaborate plant construction must be provided and that very long treatment times are required for the fibres.

The object of this invention is therefore to provide a plant and a process for a fluid separation membrane which have a higher productive output than known plants and processes while achieving unchanged or increased product quality in terms of permeability and/or selectivity.

This object is achieved according to the invention with a plant for producing a hollow fibre, wherein the hollow fibre is formed from at least one polymer solution. The plant comprises a control and regulation unit, a media reserve and supply unit, a spinning unit for forming the fibre, wherein downstream of the spinning unit for the treatment of the fibre at least the following plant units are included, in the stated sequence:

a. an exchange unit for solvent exchange, which in particular may comprise a (first) fixing unit or be formed substantially thereof, in which the fibre directly after the spinning unit can be introduced or conducted through, and wherein advantageously a washing unit can be included downstream of the fixing unit,
b. at least one aftertreatment unit, comprising
a thermal treatment unit having at least one thermal treatment station, wherein

downstream of the exchange unit at least one thermal treatment station of the thermal treatment unit is provided, which is configured as a heat treatment station comprising at least one heat treatment oven, wherein the at least one heat treatment oven comprises at least one infrared emitter (IR emitter), wherein the fibre can be at least temporarily accommodated in and/or conducted through the at least one heat treatment oven. In this

case, the heat treatment oven is designed and adjustable in such a way that the fibre which is at least temporarily accommodated and/or conductable and wherein the heat treatment oven is configured to heat the at least temporarily accommodated and/or conductable fibre for a defined time (heat treatment time) to a temperature below the glass softening temperature (T_g), in particular and also

i) in the range up to 80°C below the glass softening temperature (T_g), and/or

ii) in the range of greater than 250°C.

In an advantageous embodiment, the heat treatment oven is designed and adjustable i) to heat the at least temporarily accommodated and/or conductable fibre for a defined time (heat treatment time) up to a limiting temperature which is not more than 1°C to 4°C below the glass softening temperature (T_g), in particular to a limiting temperature in the range of 70°C to 1°C below the glass softening temperature, preferably in the range of 65°C to 1°C, and/or ii) to heat the fibre to a temperature of greater than 250°C to 450°C, preferably 280°C to 450°C.

In particular, it is advantageous here if the plant is designed to maintain the difference of 1°C to 4°C from the glass softening temperature (T_g) with regard to the fibre that is at least temporarily accommodated and/or conductable, in order not to damage the structure, in particular the desired porosity, of the hollow fibre. In order to avoid damage to the fibre owing to fluctuations in the heating temperature, it may be preferred if the plant is designed such that instead of the aforementioned 1°C difference to the T_g, a difference of 1.5°C, 2°C, 2.5°C, 3°C or 3.5°C from the T_g can be provided.

By analogy, "not more than 1°C, in particular not more than 4°C" here means that it is particularly advantageous if the difference of at least 1°C, preferably 4°C, from

the respective glass softening temperature (T_g) of the fibre material (polymer substrate) is maintained, in order not to damage the structure, in particular the desired porosity, of the hollow fibre.

In order to set defined material states of the fibre and to improve the regulatability of the heat treatment, according to an advantageous embodiment of the system, it can be provided that at least one further thermal treatment unit is arranged, comprising a drying station upstream of the heat treatment station.

The fibre is thus directly or indirectly heat-treated in the heat treatment station or heat treatment oven by the IR radiation of the at least one IR emitter. "Indirectly" here means that the IR radiation can also reach an individual fibre through adjacent individual fibres and/or via reflectors.
"Heat treatment" here means heating the fibre for a defined time (heat treatment time) up to a limiting temperature that is not more than 1°C to 4°C below the glass softening temperature (T_g), in particular in the limiting temperature range from 80°C to 1°C below the glass softening temperature, preferably in the range from 70°C to 1°C, particularly preferably in the range from 65°C to 4°C below the glass softening temperature.

The glass softening temperature T_g can be determined by various forms of differential scanning calorimetry (DSC), in particular according to the method of DIN 53765.

In an analogous manner, the upper limiting temperature and limiting temperature range employed can also be the degeneration temperature or decomposition temperature (T_d) of a fibre and/or of the fibre material (polymer substrate), depending on which effect, degeneration or glass softening, occurs earlier in the heating of the fibre and/or of the fibre material (polymer substrate).

The degeneration can, for example, comprise burning, incineration, cracking, embrittlement or other destructive effects.

In the case of a fibre material for which $T_d < T_g$ is present as a physical property, the degeneration then consisting in the fact that at $T_d < T_g$, in particular $T_d << T_g$, increased material shrinkage already ensues, the limiting temperature should be less than T_d. Thus the plant should advantageously be designed, with regard to the at least temporarily accommodated and/or conductable fibre, always in an analogous manner with the limiting temperature to maintain the difference to T_d, in order in particular to not negatively influence the fibre dimensions (macrostructure) and/or the pore structure (microstructure) of the fibre.

Fibre materials with a pronounced degeneration at temperatures from and above T_d are, for example, PIM-1 polymers with a high intrinsic microporosity. An example of a PIM is the polycondensation product of 5,5′,6,6′-tetrahydroxy-3,3,3′,3′-tetramethyl-1,1′-spirobisindane (CAS No 77-08-7) and 1,4-dicyanotetrafluorobenzene (CAS No 1835-49-0).

This degeneration temperature T_d can be determined by thermographic analysis in a nitrogen atmosphere (Konnertz, N. et al, Molecular mobility of the high performance membrane polymer PIM-1 as investigated by dielectric spectroscopy, ACS Macro Lett. 2016, 5, 528–532).

Thereby, in the case of the polymer materials in question, in particular a temperature of greater than 250°C, in particular greater than 280°C, is achieved in the heat treatment oven by means of the IR emitters, with the above-mentioned, substance-dependent difference being maintained.

The thermal treatment unit according to the invention can thus comprise a single thermal treatment station for implementing a single drying step, which is configured as a heat treatment station with a heat treatment oven and the at least one IR emitter and by means of which the fibre or the polymer material is heat-treated. In this embodiment with a single thermal treatment station, precisely speaking, a combined drying and heat treatment step takes place. However, it has emerged that the high energy input in the heat treatment oven onto an externally wet or very damp fibre leads to adverse impairments of the fibre during heat treatment. Thus the thermal treatment unit can advantageously additionally comprise at least one further thermal treatment station for carrying out at least one further drying step, arranged upstream of the heat treatment station, in particular directly upstream of the heat treatment oven. The heat treatment in the heat treatment station always represents an independent drying (step) for the fibre, owing to the high temperatures applied.

The additional thermal treatment station may advantageously comprise, or be connected to, a drying means alternative to the IR radiation and a heating apparatus alternative to an IR emitter. In particular, a drying station may be provided for an independent drying step after the exchange station for discharging the solvent of the exchange station, in particular upstream of a coating station, for producing defined surface properties of the fibre when introduced into the coating station. Furthermore, advantageously downstream of a coating station, at least one further drying station for a further drying step can be provided, in particular for removing the solvent contained in the coating station or in the coating material used therein. Even if the term "a thermal treatment unit" is presently used, this does not usually represent a structural unit, but rather

serves to refer collectively to all the thermal treatment stations and/or steps, i.e. different drying station and implementation of the respective drying steps. It is particularly advantageous if directly upstream of the heat treatment station a drying station is provided, which can be identical with one of the above, in order to produce at the start of the heat treatment station a state defined with regard to the moisture content of the fibre and thus, in particular for the high-energy heat treatment, to enable as short a duration as possible.

The "heat treatment station" is to be understood presently in particular as the central heat treatment oven and all directly associated supply and removal apparatuses, such as, in particular, gas supply conduit, gas drain conduit, sensors, optionally power controller/control unit, cooling unit with the required connections, etc. The "heat treatment oven" comprises the at least one IR emitter and can receive at least one individual fibre inside, in order to heat it at least temporarily to a temperature below the glass softening temperature T_g or the degeneration temperature T_d by means of IR radiation, as described herein. Although the heat treatment station is not identical to the heat treatment oven, in isolated cases below no distinction is made between heat treatment station and heat treatment oven; the text context reveals what is meant - for example, phrasing to the effect that "the heat treatment station comprises an IR emitter" means that "the IR emitter is included by the heat treatment station as part of the heat treatment oven".

Although the term "IR emitter" is referred to primarily below, this shall not only refer to the actual infrared radiation means (IR radiation means), but shall also include and refer to an IR unit which comprises one or more IR emitters.

A plant operating continuously and an associated continuous process have been found that make it possible to reduce the heat treatment times known from the prior art by around a factor of 0.001 or more by means of an electronic batch-operation convection oven, for example. Furthermore, protracted heating steps have been completely avoided.

In addition to the substances exemplified herein, "polymer solution" presently also refers to liquid and/or liquefied polymer melts. "Polymer solution" is especially to be understood as meaning liquid and/or liquefied polymers which are present especially in a solvent or solvent mixture.

In addition to substances exemplified herein, "polymer solution" presently also refers to liquid and/or liquefied polymer melts. "Polymer solution" is especially to be understood as meaning liquid and/or liquefied polymers which are present especially in a solvent or solvent mixture. The polymer solutions include:

- aromatic polyimides, aromatic polyetherimides, aromatic polyamides, polyarylene ethers, aromatic polybenzimidazoles and/or mixtures thereof;
- polysulfones and/or mixtures containing polysulfones;
- solutions of polymers having intrinsic microporosity (PIMs);
- generally, solutions of polymers suitable for the production of membranes for gas separation, in particular for the production of (spun) hollow fibre membranes;
- cellulose-based solutions suitable for the production of membranes for gas separation.

The polymer solution may further comprise 2D or 3D fillers, which are suitable in terms of size and constitution for spinning using the spinning unit for

hollow fibres. These fillers can advantageously be, for example, zeolites, MOF particles (Metal-Organic Framework materials), mexenes (MXenes) and/or graphenes. Furthermore, hollow fibre membranes are to be included in which the active separation layer is formed at least partially of a 2D or 3D filler, such as zeolites, MOF particles (Metal-Organic Framework materials), mexenes (MXenes) and/or graphenes.

Particularly in the presence of fillers in the polymer solution, it is advantageous if individual fibres or smaller (partial) fibre bundles of up to a maximum of 180 individual fibres are heat treated, ideally up to a maximum of 120 individual fibres, ideally up to a maximum of 60 individual fibres per (partial) fibre bundle. Without being limited to one interpretation, fillers can cause greatly increased absorbance and/or reflection of the energy, especially of IR radiation, so decreasing the homogeneity of the heat treatment of the individual fibres in the fibre bundle.

In the plants, the fibres are preferably conveyed as a strand and uncut, with the drive conveying via conveyor rollers and/or at least one traction apparatus in the main conveying direction. The main conveying direction represents the superordinate direction of movement in the x direction. Advantageously, the fibres are diverted from the main conveying direction in the x-direction on partial sections many times, in particular in the z-direction, for example in the washing unit, the thermal treatment unit, etc., in order to enable the required residence time in the respective unit.

The individual fibres, at least initially distanced from one another, are kept at a distance by comb-like or finger-like arraying elements of arraying units; usually, the individual fibres are arranged in an alignment next

to each other, i.e. distanced in the y-direction from each other.

"Fibres" are to be understood presently as the group of individual fibres, regardless of the plant location or the treatment type, unit and/or station, with the plant location and treatment type, unit and/or location being apparent from the context. In particular, "fibre(s)" should primarily be understood to mean the uncut (individual) fibre(s) or the fibre strand. The terms "fibre" and "individual fibre" are sometimes used synonymously.

The term "bundle", "partial bundle" or "(partial) fibre bundle" refers presently to an arrangement of a plurality of individual fibres in which the individual fibres are arranged such that they touch each other and, for example, are substantially parallel to each other or twisted relative to one another around a common longitudinal axis. A "bundle", "partial bundle" or "(partial) fibre bundle", hereinafter referred to for short as "bundle", may be held and/or arrayed by a guiding and/or gripping tool, and may only be arrayed temporarily, unless something to the contrary is expressly described. Furthermore, a "bundle" may be mutually fixed and arrayed at least uni- or bilaterally through a fixing element, such as a band, a laced fastening or a clip, for example, at least temporarily.

The "main conveying direction" is determined by the sequence of the treatment units and stations of the plants, with the fibres themselves being diverted by means of guide elements as and when required. Thus the distance-dependent thread direction in the direction of the longitudinal fibre axis may be different from the (higher-level) main conveying direction. In the present case, the respective local "transport direction" within the plant is considered in particular parallel to the

orientation of the "longitudinal fibre axis"; local, lateral displacements for changing the (transverse) distances of fibres to each other may be included here.

Advantageously, the plant comprises a multiplicity of guide and drive elements along the thread conveying section, which, for example, may be guide fingers, guide rollers, baffles or other suitable elements, where guide and drive elements may be located inside and outside of the treatment unit, including within liquids or within a volume of liquid.

The media included in the media source and feed unit may include
- fibre media, namely media and materials from which the fibre itself is formed at least temporarily, and
- process media required for other treatment of the fibre and/or of plant elements. These may be, for example, the core fluid for forming the hollow fibre, fixing media for the raw fibre, washing liquids, such as water, alcohols, air, gases (N2), etc., or other consumable media.

The spinning unit advantageously comprises a plurality of spinning heads, the spinning heads being configured in a manner known in principle and being connected to the corresponding supply lines for media, in order to form hollow fibres and discharge them into a fixing unit or a fixing medium.

The control and regulation unit can be configured in one or more parts and is configured to operate the plant in at least partially automated fashion. Modification of operating parameters and/or the display of plant-relevant information can be done via a central cockpit and/or decentralized input devices.

Even when treatment units, such as the "spinning unit", "fixing unit", "washing unit", etc., are stated presently

only in the singular, they can also be constructed in several parts and/or occur several times along the main conveying section. Thus, for example, the generally known washing unit may be formed of two or more washing stations or washing tanks, which are filled with identical or different (washing) media and/or whose (washing) media have different physical properties, such as temperature, flow velocity, flow direction, etc. Advantageously, the washing liquids are water-based and/or organic washing liquids.

Aside from the heat treatment device and the heat treatment step, with regard to the plant details and the process details, reference is also expressly made to WO 2014/202324 A1, the content of which in this respect is expressly made the subject of this application, as an exemplary embodiment of the plant and the process, insofar as a hollow fibre is produced from a polymer solution. This applies, for example, to the media used and the treatment units and their identical or analogous structure, such as, for example, the spinning unit, fixing unit (called "precipitating bath" there), washing unit and/or thermal treatment unit. The fixing unit is referred to in WO 2014/202324 A1 as a "precipitating bath", in which a solvent exchange takes place. The exchange takes place, for example, from an aprotic dipolar solvent of the polymer solution, such as dimethylformamide (DMF) and/or N-methyl-2-pyrrolidone (NMP), to a non-solvent for the polymer material, such as water, for example. In at least one subsequent washing unit, an organic solvent or mixture thereof, such as isopropanol and/or hexane, for example, then removes the non-solvent.

This also affects, for example, the fibre medium as feed for the spinning step, which is called "casting solution" in WO 2014/202324 A1.

The aftertreatment unit comprises the thermal treatment unit and hence also the heat treatment station and may further also comprise a washing unit upstream of the thermal treatment unit. The aftertreatment unit may, furthermore, comprise at least one coating station, in particular a coating station in which as coating material a polymer material, such as polydimethylsiloxane (PDMS), for example, is applied to the fibre. Application may be carried out using a bath or a spraying apparatus.

In one advantageous embodiment of the plant, it can be provided that the thermal treatment unit as a thermal treatment station, in particular as a further thermal treatment station, comprises a drying station through which the individual fibres are passed, wherein the drying station comprises

- at least one drying element and/or
- a feed conduit and a drain conduit for a drying medium, such as in particular a heated gas, and

whereby the interior of the drying station is at least at a temperature of over 30°C, advantageously of over 40°C, and ideally of over 50°C, where substance-dependent limits for explosion protection must be observed. In this case, it is also advantageous if the interior can be heated to at maximum 200°C.

In the present context, "drying station" and each "thermal treatment station" which is not described as a "heat treatment station", "heat treatment oven", "for heat treatment" or the like means a station in which no heat treatment of the fibre takes place or can take place, and thus the fibre is heated or can be heated only far below the glass softening temperature, owing to its design and/or in normal operation.

The fibres are preferably dried at a temperature in the range between 50 and 100°C, with the washing medium, in

particular ethanol, isopropanol, butanol, heptane and hexane, being drawn off.

The drying station is ideally shaped such that the total moisture content after drying is 0 to 5% by weight, preferably < 3% by weight and especially preferably 0.1 to 3% by weight. The moisture content is essentially or exclusively formed from the fractions of water and the respective solvent.

Advantageously, the fibres are conducted as a strand, i.e. uncut, through this treatment station and supplied downstream, in particular directly downstream of the heat treatment station.

In a further advantageous embodiment of the plant, it can be provided that the thermal treatment unit comprises as thermal treatment station, in particular as further treatment station, a dehumidification station in which the individual fibres, in particular as a strand and uncut, are passed through at a distance from one another transversely to the axial direction (longitudinal fibre axis (A_F)), which station comprises at least one nozzle element which is directed at the path of at least one individual fibre or fibres, and by means of which a gas jet can be guided onto at least one individual fibre, in particular a group of individual fibres. Advantageously, all of the individual fibres are dehumidified by means of such nozzle elements. The dehumidification in this station essentially consists of the removal of adhering, external liquid from the respective fibre fluid-dynamically by a gas pulse. The nozzle element may be in one variant embodiment a gas nozzle and/or a group of gas nozzles, which are formed as a slit nozzle.

In a further advantageous embodiment of the plant, it can be provided that

i) a grouping station with a grouping unit is arranged ahead of the heat treatment station,

ii) the heat treatment station is arranged within a grouping unit, and/or

iii) a grouping unit is arranged between two grouping stations, wherein in the grouping station and/or in the grouping unit at least one grouping means is provided by means of which at least one group of n individual fibres can be arranged in mutual contact as a bundle, wherein n is an integral value from 2 to 180, preferably from 20 to 150, ideally from 30 to 120. Groups of individual fibres with n > 180, in particular n up to 600, are also possible.

Surprisingly, bundles of up to n = 180 individual fibres, preferably up to n = 120, ideally up to n = 80 can advantageously be heat-treated simultaneously by means of the continuously operating heat treatment oven and the included IR emitters.

In an advantageous embodiment, it can be provided that the (end) bundle is formed in at least two steps, for example by forming, in a first step, in a grouping station, two or more partial bundles from the total number of the conveyed individual fibres by means of grouping means and forming the (end) bundle, with the total number of the individual fibres, downstream in a grouping station. Advantageously, the heat treatment station with the IR emitter(s) of the heat treatment oven is arranged between the first grouping station and the (last) grouping station for forming the (end) bundle. In the case of very large bundles, this can have the advantage that a more uniform and, in particular, faster heat treatment of all of the individual fibres can take place. Owing to parallel treatment of the two or more partial bundles in the heat treatment station or the heat treatment oven, the latter can be made structurally smaller.

In a further advantageous embodiment of the plant, it can be provided that the heat treatment station and/or the heat treatment oven comprises a ventilation unit and/or is connected to a ventilation unit and/or the heat treatment oven has at least one gas inlet opening, in particular a gas inlet opening for atmospheric air.

In this way, a gas supply and/or gas discharge can take place. Furthermore, escaping vapours can be selectively discharged and supplied gases, such as air and/or inert gases, e.g. N2, CO2, can be pretreated and conditioned in an appropriate manner. Thus the ventilation unit may comprise a gas conditioning unit or be connected thereto. The gas conditioning unit advantageously comprises any treatment elements for setting an advantageous gas quality, such as (HEPA) filters, dehumidifiers and/or temperature adjustment units, for example. A "ventilation unit" refers to any driven gas flow that is not caused solely by thermal conditions. The ventilation unit is advantageously connected to the heat treatment station and/or the thermal treatment unit via a feed conduit and/or a drain conduit, where the drain conduit can be connected to a gas cleaning unit, such as, for example, a gas scrubber and/or a filter, in particular an activated carbon filter, to separate off harmful gas components and/or to prepare them for disposal.

Surprisingly, the gas atmosphere in the heat treatment oven has almost no influence on the quality of the fibre, especially not on selectivity and permeance. Without wanting to being settled on one interpretation here, it is assumed that due to the very short heat treatment times, the oxidative influence of oxygen is very limited, and so the heat treatment oven can be open to the atmosphere.

In an advantageous embodiment it may be the case that

i) the heat treatment chamber of the heat treatment oven is open to the atmosphere during operation as intended, and/or

ii) there is at least one gas inlet opening for regulated (active) introduction of gas into the heat treatment chamber.

Here, "in operation as intended" means the state of the heat treatment oven, in particular a largely mechanically closed state of the heat treatment oven, in which the heat treatment of a fibre inserted in the heat treatment chamber takes place. Furthermore, "open to the atmosphere" means that a wall opening and/or a supply channel is provided through which the fibre or the fibre bundle can be introduced into the heat treatment chamber and in parallel a gas or gas mixture, in particular air, is able to flow into the heat treatment chamber.

In this case, the fibre inlet and/or the fibre outlet may be formed as an inlet or outlet opening, such as a recessing or excision of material, in a heat treatment oven provided for a batchwise or continuous operation. In an improvement of this embodiment, a lock chamber may be provided, in particular a heatable lock chamber, which is preferably heated with a heating gas to limit the entry of cold and/or unconditioned ambient air. The heating gas can be air, an O2-containing gas mixture or an inert gas, such as nitrogen (N2) or carbon dioxide (CO2). However, it has been shown that the passive entry of ambient air has no adverse effect on the fibre quality, especially not on selectivity and/or permeance.

In order to improve the controllability and regulatability of the heat treatment oven, in a further advantageous embodiment the heat treatment oven may comprise a gas supply line, in particular into the interior space comprising at least one IR emitter (heat treatment chamber), wherein a gas (or gas mixture) can

be introduced into the heat treatment oven by means of a connected or connectable ventilation station. In particular, air can be introduced into the interior of the heat treatment oven. The gas supply line here may include one or more inlet openings into the heat treatment oven. Furthermore, the heat treatment oven may comprise a gas discharge, in particular from the interior space comprising at least one IR emitter, for discharging the hot gas mixture. The gas outlet may include one or more outlet openings from the heat treatment oven.

The direct gas introduction into the heat treatment oven provides an advantageous degree of freedom for controlling and regulating the oven and the fibre can be protected directly, for example, in the event of temporary overheating of the components of the heat treatment oven, without interruption of production. All aforementioned apparatuses, elements and components include all sensors and control and regulation units required for this purpose, or are connectable or connected thereto.

In a further advantageous embodiment, the ventilation station is alternatively or additionally configured to introduce or pass an inert gas into or through the heat treatment oven, in particular nitrogen (N2). Advantageously, the ventilation station is designed to produce a largely oxygen-free atmosphere in the heat treatment oven, which may in particular be such that the O2 content is below 1000 ppm, advantageously below 500 ppm, ideally below 200 ppm.

The gas flow within the heat treatment oven and in particular in the heat treatment chamber is not limited and can follow directly along or transversely to the fibre and/or take place indirectly through internal spaces of the heat treatment oven which are delimited by components, such as reflectors or partition walls, from

the interior space surrounding the fibre. Particularly advantageous is the gas flow or flow attack parallel or largely parallel to the fibre, whereby less mechanical stress and fewer vibrations are generated in the fibre or the fibre bundle.

In one advantageous embodiment, the heat treatment oven itself has a cooling unit, in particular a cooling unit which is operated with a fluid, such as water, for example.

In a further advantageous embodiment of the plant, the IR emitter of the heat treatment oven may have a power relative to the length (m_E) of the IR emitter in metres of at least 7.5 kW/m_E, advantageously of at least 10.0 kW/m_E and ideally of at least 15 kW/m_E. Advantageously, the power of the IR emitter is not more than 100 kW/m_E, preferably not more than 50 kW/m_E, and ideally not more than 35 kW/m_E. In particular with IR emitters which can be operated at a power of 8 to 17.5 kW/m_E, a polymeric hollow fibre or a bundle of up to 100 individual fibres can be heat-treated within a time period of less than 15 s, ideally less than 10 s, and specification-compliant permeances and selectivities can be achieved.

The interior of the heat treatment oven may have one or more reflectors in addition to the at least one IR emitter in the interior space surrounding the fibre (heat treatment chamber). In this case, at least one reflector may be mounted on a wall of the IR emitter or form at least part of a wall or a wall section of the heat treatment oven.

In a further, advantageous embodiment, it can be provided that the heat treatment oven is formed of two or more parts and for this purpose has at least two mutually shielded (oven) interiors or (oven) portions. Such a heat treatment oven is designed to heat the fibre in at least one heating step in a first interior or portion for a

first treatment period with a first IR radiation power and to heat treat the fibre in at least one power step in a further interior or portion downstream for a further treatment period with a further IR radiation power, different from the first. In this case, "shielded" means that at least one separating and/or diverting element, in particular a separating element with a suitable small passage opening, is provided, that in particular the IR radiation from the further interior or portion does not reach the first interior or portions in which the heating of the fibre can be carried out. The separating element can also be a lock element or interspace without a heating element, in particular without an IR emitter.

In a further, advantageous embodiment, at least one fibre accumulator can be provided before and/or after the heat treatment station, so that the fibre or the bundle can be conveyed within the heat treatment station with a reduced conveying speed or can be stopped at least temporarily, i.e. can have a conveying speed of 0 m/s for a defined partial duration of the heat treatment. Ideally, the fibre accumulator is motor driven and/or motor controllable; particularly advantageously, the fibre accumulator is integrated in an adjacent treatment unit.

Advantageously, it is provided in the plant that the heat treatment oven and/or IR emitter has a channel- or alley-like configuration and comprises at least two walls or wall portions parallel to the local fibre transport direction, especially in the direction of the longitudinal fibre axis (A_F), where the walls or wall portions can be covered with aforementioned reflectors or formed at least partially thereof. Thus, a fibre is passed uncut as a strand through the heat treatment station or the IR emitter and heat treated during conveying in the direction of the fibre axis. The IR emitter can thus in this embodiment have an inlet opening

and an outlet opening, into which the fibre is continuously introduced and discharged. To improve the controllability and regulatability of the heat treatment step and the IR emitter, the IR emitter may comprise at least one guide and diverter, in particular a movable diverter, by means of which the fibre is diverted at least once in the region and/or within the IR emitter. It is particularly advantageous if the guide and diverter is implemented adjustably, in particular motor-adjustably, so that the residence time (heat treatment time) of a fibre or a fibre portion in the IR emitter and in particular in the region of the IR radiation can be controlled as required.

In a very economical embodiment of the IR emitter or the heat treatment station, the latter is aligned as an alley or channel with walls parallel to the transport direction, in particular to the longitudinal fibre axis, where at least one IR radiation means is rod-shaped in design and is likewise aligned parallel or largely parallel to the transport direction, in particular the longitudinal fibre axis. In principle, the position and number of IR emitters relative to the conveyed fibre are not restricted if the quality-relevant parameters, such as radiation power of the emitters, duration of fibre exposure and uniformity of the radiation power on the fibre, are observed by the skilled person.

According to a very advantageous embodiment of the plant or else, in an analogous manner, of the process, it may be provided that the plant and the process are configured to transport the fibre or the fibre bundle continuously from the spinning unit to at least the outlet of the heat treatment station. In particular, it is advantageous to continuously convey the individual fibres uncut each as a strand or at least one fibre bundle uncut as a strand from the spinning unit to and through the heat treatment station.

In this case, a "continuous" conveying or transport of the fibre is also understood to mean at least temporary intermediate storage and/or stacking in a fibre accumulator.

The at least one heat treatment station with the at least one heat treatment oven can advantageously be arranged at one of the following plant locations:

- downstream of the washing unit, wherein no (further) thermal treatment station of the thermal treatment unit is arranged upstream of the heat treatment station,
- downstream of at least one dehumidification station of the thermal treatment unit,
- downstream of at least one drying station, in particular downstream of a drying station which is arranged downstream of a coating station or application station.

Overall, the heat treatment station is used in particular for concluding heat treatment of the (externally) wet (raw) fibre or the (raw) fibre contaminated with (internal) residual moisture composed, for example, of detergent, solvent and/or exchange medium, so that the skilled person can provide a suitable installation location where the fibre arrives wet, i.e. undried or with a defined residual moisture content after passing through a drying station.

Here, a "wet fibre" means that the fibre has not yet passed through a thermal treatment station after the washing unit or that only adhering (external) fluid has been stripped, drained and/or roughly removed with a gas jet under pressure or vacuum. The tempering station is generally after and outside the washing unit.

In a particularly advantageous embodiment of the plant, an end-processing unit is arranged downstream of the heat treatment station. In particular, the end-processing unit may comprise at least one winding wheel or a bundling tool and/or an applicator. Overall, the term "end-processing unit" is intended not to be construed in a restrictive manner and to describe a unit in which any kind of temporary or permanent connection of individual fibres takes place. In particular, individual fibres or a fibre bundle, uncut, can be continuously wound up with the winding wheel until the winding wheel is fully loaded and can then be cut and subsequently cut to a defined length and/or otherwise processed. By means of a bundling tool, a temporary or permanent bundling of a (partial) group of fibres can be carried out in addition or alternatively. Here, for example, all or a subset of the uncut fibres undergo inline wrapping and, optionally, tensioning with respect to one another. For tensioning, a tape, a band or other entwining means can be looped around the fibre group and suitably fixed. The end-processing unit may be completely or partially embraced by the post-treatment unit or connected to the post-treatment station. The bundled groups of individual fibres (fibre bundles) can advantageously be cut in the region of the wrapping; this is not to be understood restrictively and the cut in the transport direction of the fibre bundle may take place ahead of, behind or through the tape, the band or the entwining means.

Alternatively, the end-processing unit is arranged upstream of the heat treatment station, where the heat treatment station comprises at least one heat treatment oven with at least one IR emitter.

Another advantageous embodiment of the plant provides that a fibre accumulator (unit) is provided, also called "band store". The latter may in particular be arranged upstream of the heat treatment station. This fibre

accumulator is advantageously equipped with movable diversion elements, which are motor drivable and/or controllable and regulatable. In this way it can be made possible that fibres are produced continuously upstream of the heat treatment station and downstream of the fibre accumulator the fibres or the (fibre) bundle can be taken off discontinuously and/or at different speeds. In particular, the fibre accumulator can be used to stop the fibre and/or the fibre bundle in the heat treatment station.

In a further advantageous embodiment, the end-processing unit comprises a cutting apparatus for the fibres or the fibre group. In particular, a cutting apparatus by means of which the conveyed fibres or fibre group can be cut inline. For this purpose, it is particularly advantageous if the bundling tool and/or the cutting apparatus can be moved and/or swivelled in the fibre conveying direction in order to avoid interrupting fibre production. The end-processing unit may further be designed to form a (end) bundle of y x n fibres or the y partial bundles from a plurality of y partial bundles each of n fibres and to fix them at least temporarily.

"Temporarily fixing" here means that the fibres of such a group are fixed against each other in such a way, e.g. braced, that they can be transported and/or further treated as a group and that the falling-out of an individual fibre is reliably prevented. The formation of such (partial) or (end) bundles from a plurality of fibres is typically followed by adhesive bonding (potting) and finishing, i.e. formation of the filter cartridge, or by manufacture of the filter module with insertion of the filter cartridge.

In another advantageous embodiment, the post-treatment unit of the plant comprises a discharging station. This is used in particular at the entrance of a drying unit

or at the entrance of an end-processing unit, to diminish disruptive electrical charges; for this purpose, for example, a spray medium containing an electrolyte and/or an iron-containing substance is applied to the fibres. In an alternative embodiment, the electrical discharging is effected by contacting, in particular by means of a looping contact, with a grounded electrical conductor.

In a further advantageous embodiment, the post-treatment unit of the plant comprises a coating station, the coating station being for applying at least one coating to the separatory layer of, for example, a polymer material to the base fibre, wherein the separatory layer with the coating and the entire fibre or the at least one fibre bundle must subsequently be subjected to heat treatment in the heat treatment oven in order to set the desired separation properties of the hollow fibre membrane.

In one very advantageous embodiment, polydimethylsiloxane (PDMS) can be applied as coating material in the coating station (coating step), e.g. by means of a spraying apparatus or by means of a dip bath. In the case of an ISA membrane, the PDMS coating is used, for example, to close surface defects in the separatory layer, on whose separation properties it may also have a minor effect. A fibre thus coated is then advantageously dried in a drying station and subsequently heat treated downstream, where in the heat treatment step by means of IR radiation in total the desired separation properties of the hollow fibre are developed. Ideally, a drying station is attached to each coating station.

The present hollow fibres for gas separation may especially be of two basic types:

i) ISA membranes

These comprise a support structure and a thin outer layer as the separatory layer, wherein the

support structure and the outer separatory layer are produced monolithically from the same material in the spinning process and specific high separation properties are formed in the heat treatment step by means of IR radiation. A first separatory layer is formed during exchange (displacement) of the solvent in the fixing station of the exchange unit since displacement of the solvent results in formation of a compressed layer like a skin on the outside of the membrane. The thickness of this separatory layer is adjustable for example via the distance of the spinning nozzle from the fixing bath. In a further, advantageous embodiment it may be provided that the separatory layer or the fibre comprising the stated separatory layer is coated (coating step) with at least one polymer material distinct from the base fibre, especially with the polymer material polydimethylsiloxane (PDMS).

ii) Asymmetric composite membranes (AC membranes) comprising a base fibre as a basic or support structure and an applied outer separatory layer, wherein at least one further coating layer may be included.
To this end a porous base fibre which, in contrast to the ISA membrane, does not yet comprise an outer separatory layer is spun in a spinning apparatus and the subsequent exchange steps. This base fibre is then coated in an application station (coating application step) with at least one material distinct from the base fibre, as the outer separatory layer. This separation layer can comprise or consist essentially of

i) a polymer material corresponding to one of the polymer solutions described herein, such as aromatic polyimides, aromatic polyetherimides,

aromatic polypolyamides, polyarylene ethers, aromatic polybenzimidazoles and/or mixtures thereof, wherein optionally at least one 2D or 3D fillers may be included, such as zeolites or metal-organic framework materials, or

ii) be substantially formed of an inorganic material, such as at least one zeolite and/or at least one metal-organic compound, wherein suitable binders may be provided.

The base fibre and/or the separatory layer of an AC membrane according to i) or ii) may be provided with a coating, in particular polydimethylsiloxane (PDMS), analogously to the ISA membrane.

In one variant embodiment of the plant and the process, at least one application station or coating application step for applying a separatory layer to a base fibre may therefore be provided, to produce an AC membrane. In addition, at least one coating station may be provided, as set out above.

According to the invention, the two aforementioned basic types of a hollow fibre membrane are finally heat treated in the heat treatment oven by means of IR radiation, where the desired high separation properties are developed.

Advantageously, a drying station is located directly downstream of the at least one application station and/or the at least one coating station; in particular, a drying station is provided directly downstream of each application station and/or coating station.

The invention further comprises a process for producing a fibre which is spun from a polymer substrate, wherein

the fibre, in particular, is a hollow fibre for gas separation, and wherein the process comprises the following steps:

- step of spinning the fibre from the polymer solution,
- exchange step for the replacement of a solvent, in particular an aqueous solvent, wherein the exchange step may comprise at least one fixing step with a liquid fixing medium and/or at least one washing step in/with a washing medium, in particular in a basin with a washing medium, where the washing step may in particular be subsequent to the fixing step,
- at least one drying step, comprising a heat treatment step, in which the fibre is heated to a temperature below the glass softening temperature of the polymer substrate, wherein the fibre is heated, i.e. heat-treated, in the heat treatment step by means of at least one continuously operated heat treatment oven (251) through IR emitters.

In this case, as already described above for the plant, in an advantageous version of the process, it may be provided that the fibre is heat-treated or heated in such a way during the heat treatment step, by the fibre for a defined time (heat treatment time), also called heat treatment time, being heated to a temperature below the glass softening temperature (T_g), in particular and also
i) in the range up to 80°C below the glass softening temperature (T_g), and/or
ii) in the range of greater than 250°C.

In a further advantageous embodiment of the process, it may be provided, in the heat treatment step, for the fibre to be heat-treated or heated in such a way that the fibre for a defined time (heat treatment time)

i) is heated to a limiting temperature which is not more than 1°C, in particular not more than 4°C below the glass softening temperature (T_g), in particular to a limiting temperature in the range of 70°C to 1°C below the glass softening

temperature (T_g), preferably in the range of 65°C to 1°C, and/or

ii) to heat the fibre to a temperature of greater than 280°C to 450°C and below the glass softening temperature (T_g).

Here it is particularly advantageous if the difference of not more than 1°C to 4°C from the glass softening temperature (T_g) is maintained in order not to damage the structure, in particular the desired porosity, of the hollow fibre. In order to avoid damage to the fibre owing to fluctuations in the heating temperature, it may be preferred if instead of the aforementioned not more than 1°C difference to the T_g, a difference of not more than 1.5°C, 2°C, 2.5°C, 3°C or 3.5°C to the T_g is provided. The respective maximum temperature difference to the T_g can be provided depending on the respective fibre material and on the plant and/or the process.

By analogy, "not more than 1°C, in particular not more than 4°C" here means that it is particularly advantageous if the difference of at least 1°C, 1.5 °C to for example 4°C, from the respective glass softening temperature (T_g) of the fibre material (polymer substrate) is maintained, in order not to damage the structure, in particular the desired porosity, of the hollow fibre. This applies in the same way to the degeneration or decomposition temperature (T_d).

Even if a single fibre is mentioned in the present case, a group of single fibres or a fibre bundle can also be treated in an analogous manner. In particular, in one process variant, a fibre bundle of n individal fibres of up to n = 600 is treated by means of the IR radiation, as described above. It may be advantageous here if the individual fibres in a fibre bundle are wound, turned or twisted relative to each other with respect to a common

(theoretical) central longitudinal axis. This has the advantage that the individual fibres come into closer contact and are thus more densely packed and that the risk of fibre breaks at individual fibres is reduced during shrinkage processes.

In a preferred process variant, in the heat treatment step, the fibre can be treated by means of the IR emitter with an IR radiation power based on the emitter length in metres (m_E) of at least 5.0 kW/m_E, preferably at least 7.5 kW/m_E and ideally at least 10 kW/m_E. It may further be advantageous here if the IR radiation power is not more than 100 kW/m_E, advantageously not more than 50 kW/m_E and ideally not more than 35 kW/m_E.

All aspects and advantages described herein in respect of the plant shall also apply identically or analogously to the process and vice versa, provided they are not technically precluded/impossible.

Advantageously, the heat treatment step takes place in an inert or largely inert atmosphere in which the O_2 content in the heat treatment oven is below 1000 ppm, advantageously below 500 ppm and ideally below 200 ppm. The IR radiation is advantageously introduced by the IR emitter at least unilaterally in the direction of the fibre or the fibre bundle in the heat treatment oven and advantageously distributed by reflection from at least one reflector within the heat treatment oven.

As an outstanding effect, it was surprisingly found that the entire heat treatment time using an IR emitter for a bundle of up to 80 individual fibres requires only, for example, 0.1% of the heat treatment time, compared to a usual heat treatment oven, such as an air circulation or convection oven. According to an advantageous embodiment of the process it can thus be provided that the heat

treatment time is in the range of 0.1 to 60 s, preferably 0.5 to 30 s, ideally 1 to 20 s.

A further process embodiment may involve the heat treatment step consisting of at least two partial steps, namely a
1) heating step, in which the IR radiation power is in the range of 5% to 25 % of the maximum IR radiation power, advantageously 7.5% to 15%, ideally 10 %; and a
2) power step in which the defined maximum IR radiation power is irradiated by the IR emitter of the heat treatment oven.

The duration of the heating step is advantageously 1/6 to 1/3 of the power step, where the power step is advantageously in the range from about 0.1 to 60 s, preferably 0.5 to 30 s, ideally 1 to 20 s.

Even if a distinction is made presently between "heat treatment step" and "power step", "heat treatment step" is also used in some cases synonymously for the "power step", in which the maximum radiation power is irradiated by the IR emitter.

In an advantageous embodiment of the process,

- the liquid polymer solution and/or
- the fibre spun from the polymer solution, or the substrate,

after fixation and/or after at least one thermal treatment station of the thermal treatment unit, can have an absorbance E of less than 0.5; advantageously of less than 0.3; ideally of less than 0.2; where the absorbance E is defined as the quotient $E = \lg (l_{in}/l_{out})$ with l_{in} as the intensity of the incident IR radiation and l_{out} as the intensity of the outgoing IR radiation.

The absorbance of the IR radiation here is undertaken depending on the wavelength and the layer thickness.

The absorbance of the liquid or re-liquefied polymer solution can be measured depending on the wavelength range by means of a Vis spectrophotometer, e.g. UV-VIS spectrometer Specord 210 plus from Analytik Jena, and/or an IR spectrometer, such as the FT-IR spectrometer Vertex 70 from Bruker Corporation. Liquid polymer samples were inserted into a cuvette of the respective spectrometer. A solid sample (film piece) was inserted into a respective connected sample holder of the abovementioned spectrometers and measured. The wavelengths here were partially limited by the respective spectrometer and, if necessary, additionally resolved by a detector in the sample holder. The corresponding sample holder for solids in the UV-VIS spectrometer is a so-called integrating sphere from Analytik Jena; the one in the FT-IR spectrometer Vertex 70 is, for example, the Integration Sphere from Bruker.

The VERTEX 70 spectrometer was used to measure the spectral range from 10 cm-1 (far IR/THz range), the middle and near IR range up to the visible/UV spectral range (28 000 cm^{-1}).

To measure the solid substrate, film pieces were produced. For this purpose, 25% polymer solution (e.g. containing polyimide) with the solvent used in production was applied to a glass plate and brought to a defined layer thickness of 0.5 mm with a doctor (casting knife). Subsequently, the solvent was evaporated under an inert atmosphere in an oven at around 80°C for at least 60 minutes. The solidified film pieces were then removed from the glass plate with water and dried again. The thickness of the solidified film pieces ranged from 30 µm to 120 µm.

The film piece thus produced was inserted into the respective sample holder and the absorbance or absorbance coefficient of the transmitted beam through the film

piece was detected with the respective spectrometer in a wavelength-dependent manner.

It has proven to be particularly advantageous if the absorbance (IR absorption) of the fibre or an analogous polymer substrate (solid) or the polymer solution (liquid) for IR radiation is in the range from 0.6 to 1.8 µm, ideally in the range from 0.75 to 1.75. In other words, it is advantageous if, in particular, the solidified non-heat treated polymer substrate is largely transparent to the stated wavelengths of the IR radiation, so that even for a fibre bundle a very good, homogeneous heating of all fibres is made possible. In particular, the wavelength range of the respective maxima of the IR radiation between 800 nm and 2000 nm, with power of the IR emitter based on the length of the IR emitter from 2 to 20 kW/m_E is preferred.

Surprisingly, it could be observed that because of the aforementioned low absorbance in the wavelength ranges stated, a large group of individual fibres can also be heat treated as a (dense) bundle, while nevertheless as individual fibres exhibiting very homogeneous separation properties in terms of permeance and selectivity. In the present tests, groups of up to 100 individual fibres were successfully heat treated as fibre bundles by means of IR radiation, which achieved the required specification analogously to the conventionally heat treated fibres.

In a further process embodiment, an advantage can be that the fibre is conveyed at least for a partial duration of the heat treatment step. Advantageously, the fibre is conveyed during the entire heat treatment step.

According to a further-improved process variant, it can be provided that the heat treatment oven

i) is open to the atmosphere, in particular has an inlet or outlet opening for the fibre and a

passive gas or air entry with the conveyed fibre takes place in the heat treatment oven, and/or

ii) comprises or is connectable to a ventilation station, via which a gas (mixture), in particular air, N2 and/or CO2, is introduced into the interior (heat treatment chamber) of the heat treatment oven.

In alternative embodiments of the process, as described above for the heat treatment oven, gas routing is carried out via locks.

Thus, in a further advantageous embodiment of the process, it may be provided that the heat treatment oven is open to the atmosphere. In this case, atmospheric gas (air) in a for a continuous process can be conducted via the fibre inlet and/or the fibre outlet as an inlet or outlet opening. In a simple process variant, no lock chamber is provided. In an improvement of this embodiment, a lock chamber may be provided, in particular a heatable lock chamber, via which a first temperature control of the fibre and shielding of the interior space of the heat treatment oven takes place. Preferably, a heating gas is introduced into the lock chamber in order to limit the entry of cold or strongly temperature-fluctuating ambient air or to reduce the thermal load on the fibre at the outlet. Alternatively, the lock chamber may include a heating element. In this case, the heating gas can be a gas mixture, such as air, a gas mixture containing O2. Alternatively or in addition, the gas (mixture) may comprise at least one inert gas, such as nitrogen (N_2) or carbon dioxide (CO_2).

To improve the controllability and regulatability of the production process, a gas feed can be applied in a further advantageous embodiment, in particular into the interior comprising the at least one IR emitter, wherein by means of a connected or connectable ventilation station into

the heat treatment oven a gas mixture, in particular air, is introduced into the interior by means of, for example, a compressor (actively). The gas feed line can be applied via one or more inlet openings into the heat treatment oven, wherein a gas outlet is provided.

The direct gas introduction provides a degree of freedom for controlling and regulating the oven for the process and effectively protects the fibre in the event of, for example, temporary overheating of the components of the heat treatment oven, without interrupting the process.

Although presently the plant and the process are primarily described for continuous heat treatment, the present invention is not limited to this. The IR emitter can also be a batch unit, so that the fibres, a fibre bundle or a group of fibre bundles are heat treated in batch operation by means of at least one IR emitter. For this purpose, the fibres are advantageously cut to a desired length before heat treatment and/or advantageously fixed at least temporarily as (partial) bundles, analogously to the plant treatment units described herein. For batchwise heat treatment, it is advantageous overall to heat treat the individual fibres or the fibre bundles hanging vertically in the axial direction, so that the individual fibres remain aligned largely parallel to each other.

Absorption is a measure of the attenuation of the intensity of light or radiation, including IR radiation, by adsorption or scattering after the beam has crossed through matter. The absorption is carried out by means of spectrometers.

The solution according to the invention is described below in detail with reference to exemplary embodiments. In the figures:

Fig. 1 shows the plant in a first variant embodiment,
Fig. 2 shows the plant in a second variant embodiment,
Fig. 3 shows the plant in a third variant embodiment,
Fig. 4 shows the plant in a fourth variant embodiment,
Fig. 5 shows the plant in a fifth variant embodiment,
Fig. 6 shows two variant embodiments for the transport of the fibre in the heat treatment station,
Fig. 7 shows the test setup as a schematic representation,
Fig. 8 shows a graph comparing the temperature profiles from the tests with the known heat treatment,
Fig. 9 shows a graph of the absorbance against the IR spectral radiation of an emitter, and nine characteristic curves,
Fig. 10 shows four graphs in two partial representations I., II., which contrast a comparison of bundles of 10 and 80 individual fibres with regard to permeance and selectivity,
Fig. 11 shows two graphs for different fibre bundles, as normalized permeance and selectivity relative to the radiation energy introduced, and
Fig. 12 shows a graph of comparative measurements for the transmission.

The plant 100 as shown in Figure 1 has a main conveying direction A which is shown from left to right and in a cartesian system represents the x-direction. The plant 100 comprises a control and regulation unit 130, a reserve and supply unit 140, a spinning unit 106, a fixing unit 150, a washing unit 160 and a post-treatment unit 170. The post-treatment unit 170 comprises a thermal treatment unit 200, with a drying station 210 of the heat

treatment station 250 according to the invention, and an end-processing unit 180. The fibre 110 is transported through the plant 100 in the main conveying direction A and deflected and diverted multiply in the y-direction or z-direction in partial sections. The control and regulation unit 130 is connected to the plant 100 or components thereof via data conduits 132, as shown by way of example by a dashed conduit.

Other elements for energy and data transmission and automation of such a plant 100 are basically known to those skilled in the art and are not further illustrated here. The reserve and supply unit 140 is also shown only in sketched form and comprises tanks 142 or a tank farm for media, in particular for the polymer solution 102 from which the fibre 110 is formed and process media 104, a pump (unit) 146 and an (inert) gas reserve. These are connected to the spinning unit 106 in a media-conducting manner. The fibre 110 is formed in the spinning unit 106 in an otherwise unspecified manner, as described, for example, for a hollow fibre from a polymer solution in WO 2014/202324 A1. Alternatively, the fibre can also be obtained from a polymer melt, in particular as hollow fibre. The process media 104 here are in particular the process media required in the spinning process, such as the core fluid for forming the inner cavity of the respective individual fibre 110.

The freshly formed fibre 110 is introduced into the fixing medium, a non-solvent, of the fixing unit 150 and is discharged via guide rollers 108 from the tank of the fixing unit 150, where alley guides 109 which have a comb-like or multi-finger-like configuration orient the individual fibres 110 in the y-direction at a defined distance from one another. The washing unit 160 comprises a plurality of guide rollers 108 which are arranged below the fluid level of the washing liquid and by means of which the fibres 110 are held in the washing liquid, in

particular in an organic washing liquid, for a defined cycle time to remove the non-solvent from the fibre 110. In a manner not shown, the washing unit 160 may comprise a plurality of serially arranged tanks.

Connected downstream of the washing unit 160 is the drying station 210, where in a grouping station 260 at the entrance to the drying station 210 the fibres 110 are combined into a bundle 112 and passed as a strand via diversion rollers 108 through the drying stations. The drying station 210 is connected to a gas feed conduit 212 and a gas drain conduit 214, where in the gas feed conduit 212 there is a heat exchanger 216 for heating the feed conduit C. The feed conduit C is, in particular, dehumidified air. In the drying station 210 the bundle 112 in the form of a strand is repeatedly deflected by deflection rollers 108 while it is surrounded and dried by the heated gas. The deflection rollers 108 are typically static bodies over which the conveyed fibre 110 or the bundle 112 is drawn, where in certain embodiment variants (not shown) it may be advantageous to drive at least individual deflection rollers 108 such that they rotate about their own longitudinal axis and in the direction of the longitudinal fibre axis A_F and thus reduce the stress within the fibre 110 or bundle 110.

The heat treatment station 250 connected in the main conveying direction A comprises a heat treatment oven 251 as a central element with an IR emitter 252 and a reflector 254, both of which are arranged in a housing 256 of the heat treatment oven 251. The heat treatment oven 251 is configured as a channel and receives on the input side continuously the predried bundle 112 for a heat treatment time (throughput time) of about 10 s, where the heat treatment time can be divided into two or more component steps, such as a heating step and a power step.

The heat treatment station 250 or the heat treatment oven 251 is connected to a gas feed unit 270 and to a gas drain unit 272, where a compressor 226 and a gas filter unit 274 are parts of the gas drain unit 272. The gas feed and drain 270, 272 are part of a ventilation unit, where the gas feed conduit 270 is connected to an inert gas source. At the discharge end of the heat treatment station 250, opposite to the input side, a conveying means 280 is arranged, which is configured as an arraying and conveying unit and which transmits the (fibre) bundle 112 in compact formation to the end-processing unit 180. Within the drying station 210, the bundle 112 can be guided by corresponding deflections 108 vertically (z direction) in portions (several times) or, as for example in Fig. 3, horizontally (x-direction) (several times). In this case, "vertical" or "horizontal" means the main orientation for the purpose of linguistic illustration and also includes (slightly) inclined orientations.

The end-processing unit 180 comprises a fixing station 182, a cutting station 184, a lifting unit 186 and a transport element 188. Via the conveying means 280, the bundle 112 is transported to the end-processing unit 180 and there, at the free end and/or in the separation or cutting area of the bundle, is fixed with at least one otherwise unillustrated fixing means, such as a band, a tape, a cord, etc., as a group (fibre bundle), in particular being multiply wrapped and thus fixed as a group. Parallel or subsequently, the bundle 112 is captured by the lifting unit 186, which in the exemplary embodiment illustrated is configured as an x-y-z lifting and gripping unit. In the cutting station 184 and a separating means symbolically represented as a knife blade, a bundle 112 is cut off at a set length from the (bundle) strand, and may optionally be temporarily stored or stacked as an end-processed bundle 112.

Subsequently, by means of the transport means 188, the end-processed bundles 112 are passed in transmission direction B out of the end-processing unit 180.

The plant 100 shown in Figure 2 differs from the plant of Fig. 1 in particular in that no drying station 210 is provided, but the fibres 110 after the washing unit 150 are directly introduced into the heat treatment station 250 or the heat treatment oven 251, which can be formed in principle analogously to the aforementioned heat treatment station 250 from Fig. 1. Connected downstream of the washing unit 160 is the heat treatment station 250, where in a grouping station 260 at the entrance to the heat treatment station 250 the fibres 110 are combined into a bundle 112 and passed as a strand linearly, i.e. without further diversion, through the heat treatment station 250. On the discharging conveying section and above the washing unit 160, a dehumidification station 220 is arranged, which drives liquid adhering to the fibre 110 back into the tank of the washing unit 160 by means of the nozzle units 222 in the region of the deflection roller 108 in said station.

In the dashed-framed, lower detailed illustration, the guide of the fibres 110 at the transition from the washing unit 160 to the heat treatment station 250 and the grouping station 260 there is shown in a plan view. Two further, analogous plan views are shown in Figure 6. As shown in the detailed view of Figure 2, at the exit of the washing unit 160, the fibres are spaced from one another by means of the alley guide 109 in the y-direction and compressed downstream through the lateral guide rails 262 of the grouping station 260 to form a bundle 112 of the then mutually contacting individual fibres 110.

The compaction takes place in the y-direction and in the z-direction, where in addition height guides (not shown) analogous to the lateral guide rails 262 can be provided

in order to carry out the compaction in the z-direction in a mechanically defined way and to avoid deformation of the hollow fibre geometry. For this purpose, at least two groups of the fibres to be bundled for a defined conveying section at different heights (z-direction) are guided through such height guides before they are placed on top of each other in the z-direction and shaped into a common bundle 112. This plant setup without a drying station 210 and the associated process regime has been made possible by the use of a heat treatment station 250 with an IR emitter 256, because it has surprisingly turned out that it is possible with continuous heat treatment to prevent adhesion even of initially relatively wet individual fibres 110 in the bundle 112. In other words, even relatively moist fibre bundles 112 can be heat-treated without or after only a small amount of drying by means of IR radiation.

Owing to the elimination of the drying station 210 and the associated conduits and assemblies, considerable energy savings and considerable reductions in the required footprint of the plants are entailed.

Figure 3 shows, in a very schematic manner, the plant 100 and the fibre 110, and also the bundle 112 on its way through the plant 100. In addition to or in contrast to the exemplary embodiments of Figures 1 and 2, the plant 100 comprises a coating station 120 and a discharging station 190, which are known in principle. The aforementioned grouping stations are not shown in Figure 3 and can be provided as required. Analogously to the plant 100 shown in Figure 1, at least temporary winding, stacking and/or cutting of the fibre bundles 112 takes place in the end-processing unit 180. In this plant design, differently to the above, the end-processed and cut fibre bundles 112, in particular a plurality of at least temporarily end-processed fibre bundles 112, are heat treated downstream of the end-processing unit 180

batchwise in a heat treatment oven 251 of the heat treatment station 250 by means of an IR emitter (not shown).

In the exemplary embodiment of Figure 4, unlike the plant variant of Figure 4, the heat treatment oven 251 of the heat treatment station 250 is arranged upstream of the end-processing unit 180. The mutually spaced apart individual fibres 110 or the at least one fibre bundle 112 are continuously transported through the heat treatment oven 251 and heat treated. In particular, the transport speed and/or the length of the heat treatment oven 251 are dimensioned and/or matched to each other in such a way that the required residence time (heat treatment time) of the individual fibres 110 or the at least one fibre bundle 112 in the effective zone of the IR emitter (not shown) can be maintained.

In the exemplary embodiment of Figure 5, in contrast to the previous embodiment variants of Figure 1 to Figure 4, and in accordance with the invention, the plant also comprises a fibre accumulator 230, which is arranged ahead of the heat treatment station 250 as part of the thermal treatment unit 200. Thus, an accumulation step for the fibres 110 or bundle 112 can be carried out before the heat treatment step.

The fibre accumulator 230 has two opposing groups of diversion elements 232, which, driven dynamically as a group or individually, can change their position relative to each other. In this way, a defined fibre length or bundle length can be accumulated, while e.g. downstream of the fibre accumulator 230 the fibres 110 or the (fibre) bundle 120 are transported at a slower rate, or temporarily stopped, compared to the case upstream of the fibre accumulator 230.

In other words, the plant comprises

- a first conveying section F1, in which the fibres can be conveyed at a first conveying speed v1, in particular can be conveyed continuously, and
- a second conveying section F2, in which the fibres can be conveyed at a second conveying speed v2, in particular can be conveyed discontinuously and/or with varying speed, wherein

a (dynamic) fibre accumulator 230 is arranged between and/or in the transition from the conveying section F1 and the conveying section F2.

The conveying section F1 here comprises the spinning unit 106, the fixing unit 150 and the washing unit 160, and conveying section F2 comprises the heat treatment station 250 with the heat treatment oven 251. In the area of the conveying section F1 and/or F2, a (respective) strand drive can be provided to pull the fibres 110 or the bundle 122. In particular, in the conveying section F2, the strand drive is used for the complete or partial emptying of the fibre accumulator 230 and/or for accelerating the fibres 110 or the bundle(s) 112, as an accelerating means, out of the heat treatment station 250.

In this embodiment, in particular, the storage capacity of the motor-driven fibre accumulator 230, the possible acceleration and transport speed of the fibre accumulator 230 in the accelerated removal of the stored fibres 110, 112 and the length of the heat treatment oven 251 are dimensioned and/or matched to each other in such a way that the required residence time (heat treatment time) of the individual fibres 110 or the at least one fibre bundle 112 in the effective zone of the IR emitters (not shown) can be maintained.

Figure 6 in a plan view shows two further variant embodiments of grouping stations 260 and the resulting

orientations and positions of individual fibres 110 in the heat treatment station 250. The partial view I. shows grouping station 260, which essentially consists of two comb-like or multi-finger-like alley guides 109, wherein the two opposing alley guides 109 have different distances between the individual fingers or individual guides. Both alley guides have the same number of individual fingers or the number of individual fingers depends on the number of parallel-guided individual fibres 110. In this case, an individual fibre 110 may optionally be present on both sides of individual fingers, depending on the cross-sectional area and cross-sectional geometry of the individual fingers.

The alley guide 109 arranged at the entrance of the heat treatment station 250 and of the heat treatment oven 251 approximates the individual fibres 110 to a few millimetres, for example up to 2 mm to 3 mm. There is no densification until the parallel individual fibres 110 come into contact with each other and the individual fibres 110 span essentially a common plane with their longitudinal fibre axes. Although this embodiment is structurally most complex, it allows the shortest heat treatment times for the fibres 110 and thus requires the smallest extension of the heat treatment station 250 in the main conveying direction (x-direction). Optionally, a second grouping station 260 can be arranged downstream, by means of which the (end) bundle is shaped.

The partial view II. shows the grouping station 260 (here 260.1), which also essentially consists of two comb-like or multi-finger-like alley guides 109, wherein the two opposing alley guides 109 also have different distances between the individual fingers or individual guides. The alley guide 109 arranged at the entrance of the heat treatment station 250 has a significantly smaller number of individual fingers or individual guides than the alley guide 109 at the outlet to the washing unit 160 or the

drying station 210. This is due to the fact that grouping station 260 and the introductory alley guide 109 shape multiple partial bundles 112 from the total number of individual fibres 110 and subsequently pass them as partial bundles 112 through the heat treatment station 250. In the example shown, three partial bundles 112.1, 112.2 and 112.3 are shaped, which are heat treated parallel at a distance from each other in the heat treatment station 250 and transported through this heat treatment station 250. The partial bundles 112.1, 112.2 and 112.3 can subsequently be combined in a second grouping station 260.2 to form an (end) bundle 112.

Even if the heat treatment oven 251 is presently always shown in a horizontal (recumbent) orientation in which the transport direction of the fibre 110 or the fibre bundle 112 is aligned in the direction of the x-axis or the main transport direction A, this is not to be understood restrictively. It is understood by the skilled person that the heat treatment oven 251 can assume any orientation and/or inclination in space, especially vertical or slanted. The alignment and/or transport direction of the fibres and/or the fibre bundle then follows this orientation of the heat treatment oven in an analogous manner and can also take place, in particular, from bottom to top or from top to bottom.

The laboratory and test setup shown in Figure 7 and the graphs shown in Figures 8 to 11 are described and discussed below together with the discussion of the test results.

Experiments and test results

The tests were carried out with a heat treatment station 250 as shown in Figure 7. The heat treatment station comprised as a central element a heat treatment oven 251, which has a closed housing 256 and in which an IR emitter 252 extended in the longitudinal direction.

The housing 256 had a foot element and a head element and was configured as an elongated cuboid body whose longitudinal axis was aligned perpendicular to the horizontal in laboratory operation. The IR emitter 252 was arranged off-centred on one side, and had a length of around 21 cm. The emitter 252 was surrounded by curved metallic reflectors 254, which had an extension of around 27 cm in the direction of the longitudinal axis and protruded on both sides upwards and downwards beyond the IR emitter 254. In addition to the reflectors 254 extending substantially parallel to the IR emitter 252, head and foot reflectors were present, so that in the closed state the interior of the housing 256 had a central IR radiation space and a rear space, where in the rear space substantially no IR radiation could be incident. The housing 256 was divided in the longitudinal direction and connected via hinges, so that it could be folded open for introduction or removal of individual fibres or a fibre bundle (not shown in Fig. 7).

On the head element, a fixing unit 258 was arranged, which was designed to hold individual fibres 110 or a fibre bundle 112 in a hanging manner. The individual fibres 110 or the fibre bundle 112 was clamp-weighted at the free, lower end with a weight 259, so that a constant tensile force could be imposed on the individual fibres 110 or the fibre bundle 112; in as-intended operation, the weight 259 was held in suspension at the fibres with a small distance above the foot element. The fixing unit 258 and the weight 259 were arranged in the rear space, for which purpose the reflectors in the head and foot areas had a corresponding passage opening.

Further arranged in the foot space is a lateral guide and contact surfaces for the weight, so that a fibre bundle, as and when required, can be wound or twisted one or more times about the longitudinal axis and could be heat

treated in such a wound or twisted arrangement of the individual fibres in the fibre bundle.

A gas feed conduit 270 consisting of two conduits led into the interior of the housing, via which a constant volume flow of nitrogen (N2) could be passed into the interior. The N2 volume flow was discharged - usually continuously discharged - from the inside of the housing via a central gas drain conduit 272.

The IR emitter 252 was connected to a control unit 134 for power regulation, where the control software was stored in a connected control and regulation unit 130 and operated by a processor unit there, which in laboratory operation was a commercially available PC.

The fixing unit 258 and the weight 259 were designed to fix a fibre bundle 112 of up to 100 individual fibres 110 and to keep it under constant tensile force.

The tests were carried out in batch operation. Furthermore, a closed water cooling system was arranged in the housing, and was connected to a corresponding cooling circuit.

Test procedure

The volume flow of N2 supplied as inert gas was 300 ml/min and the O2 content was < 100 ppm inside the housing 256. The following parameters were monitored by sensors:

- the N_2 volume flow of the gas feed and gas drain conduits,
- the O_2 concentration in the gas drain conduit,
- the temperature inside the housing in the headspace and in the footspace,
- the volume flow of the cooling water and
- the shrinkage of the fibres.

The weight used was 12 g per 10 individual fibres, and 40 l/h of water was passed through the water cooling system as a cooling water stream.

The exposure (heat treatment step) of the fibres to IR radiation took place in two steps:

1) a heating step and a subsequent
2) power step.

In the heating step, the IR irradiation of the fibre was carried out for 2 seconds at a maximum of 10% of the power of the IR emitter, based on the 100% of the intended subsequent power in the power step (target power); in the present case the 100% were in each case the maximum power of the emitter. In the power step, the IR irradiation of the fibres was maintained at the intended maximum power (target power) for 4 to 21 seconds.

After the heat treatment step, the housing 256 of the heat treatment station 250 was opened immediately and the fibres or the fibre bundle was removed, without adhering to a defined cooling time or cooling curve.

Surprisingly, the separation properties, such as, in particular, the permeance and selectivity of the hollow fibres and the filter modules made from them, which had been heat treated in such a short time by means of IR radiation, were in the range of the known, conventionally heat treated hollow fibres, as set out below more in detail.

Fig. 8 shows in a graph a comparison of the two heat treatment methods; in the graph shown, the input maximum emitter power is normalized to 1 (left y-axis) and the maximum temperature in the convection oven is also normalized to 1 (right y-axis) and plotted against the logarithmized time axis in seconds (x-axis). The heat treatment process according to the invention by means of

IR radiation is represented essentially on the left in the graph and as a solid line. The comparative test with a conventional electric oven (convection oven), in which the fibres were also heat treated hanging under constant tensile weighting in an inert atmosphere, is shown as a dashed line.

In the heat treatment process according to the invention, a heating step 300 for 1 s, a ramp-up 302 of the power of the IR emitter of also 1 s and a power step 304 for 4 s were maintained, where subsequently a cooling 306 took place, which resulted from the opening of the housing 256 towards the atmosphere. The ramp-up 302 or the time required for this results essentially from the device-specific delay time in order to provide the maximum IR emitter power, starting from the power level of the heating step.

In the comparative test, a preheating phase 400 of around 30 min was carried out, followed by a ramp-up phase 402 of the convection oven up to the maximum power and temperature of around 2 h, a heat treatment phase at the heat treatment temperature for around 2.0 h and a subsequent cooling phase 406 before the opening of the electric oven, for around 0.8 h.

The hollow fibres produced according to the invention are especially suitable for gas separation of CO_2, O_2, CH_4 and N_2, especially for gas separation of air or biogases.

The suitability of the fibre for gas separation is evaluated especially with regard to permeance and selectivity. The permeance is the ratio of the permeability to the thickness of the fibre wall or membrane wall and represents a measure of the gas flow through the fibre wall or membrane and is thus an indicator of the structure and permeability of the hollow fibre or of the fibre bundle.

Permeability and permeance

The gas permeabilities are reported in barrer (10^{-10} $cm^3 \cdot cm^{-2}$ cm s^{-1} $cmHg^{-1}$). The permeances of the hollow fibre membranes for gases are reported in GPU (gas permeation units, 10^{-6} $cm^3 \cdot cm^{-2} . s^{-1} . cmHg^{-1}$) or derived units. The permeance is the ratio of permeability to substrate thickness (membrane thickness) for corresponding units, for example the unit l m^{-2} h^{-1} bar^{-1}.

Permeability

Permeabilities for gases are measured by the pressure rise method. A flat film of between 10 and 70 µm in thickness has a gas or gas mixture applied to it on one side. On the other side, the permeate side, a vacuum (about 10 - 2 mbar) prevails at commencement of the experiment. The pressure rise on the permeate side over time is then recorded.

The permeability of the polymer (substrate) may be calculated according to the following formula:

$$P = \frac{V_{dead} . MW_{gas} . l}{\rho . R . T . A . \Delta p} . \frac{dp}{dt} . 10^{10}$$

P Permeability in Barrer (10^{-10} cm^3(STP) cm) / (cm^2 s cmHg)
[Barrer] = 1.333*1017 * [Nm^3*m/(m^2*s*Pa)]
with STP (Standard Temperature and Pressure): is 0 °C, 101 325 Pa

V_{dead}	volume of permeate side in cm^3
MW_{gas}	molar mass of gas in g mol^{-1}
I	thickness of film in cm
ρ	density of gas in g cm^{-3}
R	gas constant in cm^3 cmHg K^{-1} mol^{-1}
T	temperature in kelvin
A	area of foil in cm^2
Δ p	pressure difference between feed and permeate side in cmHg

dp/dt pressure rise per unit time on permeate side in cmHg s^{-1}

If the permeance of hollow fibres is measured, a volume rise method is used. The permeance P/I (since the thickness of the separating layer is unknown) is calculated according to the following formula:

$$P/l = \frac{Q(STP)}{R.T.A.\Delta p}.10^6$$

P/I permeance in GPU (gas permeation units. 10^{-6} $cm^3 \cdot cm^{-2}$ s^{-1} $cmHg^{-1}$)

Q gas flow of permeate side in cm^3 (STP)/s

R gas constant in cm^3 cmHg K^{-1} mol^{-1}

T temperature in kelvin

A external surface area of hollow fibre in cm^2

Δ p pressure difference between feed and permeate side in cmHg

dp/dt pressure rise per unit time on permeate side in cmHg s^{-1}

The selectivities of various gas pairs are pure-gas selectivities. The selectivity between two gases computes from the ratio of the permeabilities:

$$S = P_1 / P_2$$

S ideal gas selectivity

P_1 permeability or permeance of gas 1

P_2 permeability or permeance of gas 2

Tests V1 to V3

Tables 1a and 1b show the comparison test (V1) and two tests according to the invention with two nominally differently powerful IR emitters (V2, V3). The heat-

treated fibres were taken from an electric convection furnace (V1) of an existing production process (comparison test). These were compared with fibre from the aforementioned experimental or laboratory device (V2, V3), which were heat-treated by IR emitter in the batch process. In each of tests V2 and V3, a bundle of 80 individual fibres was heat treated in a batch run using IR radiation.

Table 1a: Test conditions of tests V1 to V3 (HT – heat treatment step)

Tests	**Type of oven**	**IR emitter nominal power [kW]**	**Number of fibres in the fibre bundle**	**Power at HT [kW]**	**Duration HT [s]**	**Energy Total [kJ]**
V1	Convective	-	>80	-	-	-
V2	IR	2.1	80	1.55	4	6.2
V3	IR	2.8	80	1.34	4	5.4

Table 1b: Test results of tests V1 to V3.

Tests	**Normalized permeance [-]**	**Normalized selectivity [-]**	**Radial shrinkage [%]**
V1	1	1	4.1%
V2	1.68 ± 0.22	1.03 ± 0.01	2.4%

V3	1.41 ± 0.50	1.11 ± 0.15	2.4%

The permeance and selectivity here were normalized to the values of the comparison test V1, i.e. the conventional heat treatment process. It emerged that an improved permeance and a constant or slightly improved selectivity could be achieved by the heat treatment process according to the invention. Furthermore, it was observed that the fibres heat treated by IR radiation showed a significantly lower radial shrinkage. The stated radial shrinkage here is set as the ratio of the radius of the dried and non-heat treated fibre (100%) to the heat treated fibre. The radii of individual fibres or a group of individual fibres were measured by means of a specifically calibrated light microscope in the laboratory.

The absolutely surprising and unforeseeable outcome was that the duration of the heat treatment process could be reduced from about 6 h to a duration of less than 10 s with an IR heat treatment, with the same and in some cases improved separation performance, permeance and selectivity of the fibres.

Test V4 (Fig. 9)

In nine characteristic curves 310 to 328, the curve set of Figure 9 shows the spectral radiation spectrum of a 3.1 kW IR emitter (nominal power) with a length of 0.2 m_E for different powers of the IR emitter. The characteristic curves differ in terms of the powers, which were reduced in steps by 10% starting from the outer characteristic curve 310.

At each wavelength from 0.25 to 5 µm, polyimide substrate was measured by VIS spectroscopy or NIR spectroscopy depending on the wavelength (curve 350). The absorbance is plotted against the y-axis in the range from 0 to 1,

where the value 1 describes the full absorbance and the value 0 describes the full transmittance or transparency for the light in the respective wavelength range (x-axis).

The IR emitter powers were measured for the outermost characteristic curve 310. The other eight characteristic curves 312 to 328 show the power of the IR emitter reduced by 10% in each case, as follows:

curve 310	1.0 * 3 kW
curve 312	0.9 * 3 kW
curve 314	0.8 * 3 kW
to	
curve 326	0.3 * 3 kW
curve 328	0.2 * 3 kW

Curve 330 shows the position of the maximum in characteristic curves 310 to 328, which follows Wien's law. It emerged that for all characteristic curves 310 to 328, the polyimide substrate for IR radiation, especially in the wavelength ranges of the maxima of characteristic curves 310 to 328, is quasi-transparent and only had an absorbance of less than 0.4.

In this case, the absorbance is defined as the ratio of emitted to transmitted IR radiation of a wavelength or in a wavelength range, as set out hereinabove.

The choice of a polymer substrate with such low adsorption properties for IR radiation in the range from 0.6 to 1.8 µm, ideally in the range from 0.75 to 1.75, has the great advantage that a large group of individual fibres can be heat treated as a (tight) bundle while nevertheless having very homogeneous separation properties in terms of permeance and selectivity. In the present tests, groups of up to 100 individual fibres were

successfully heat treated as fibre bundles using IR radiation.

Figure 12 shows the measured values of the transmission by comparison of an isotropic film and a membrane made of the same polyimide polymer substrate. The graph is plotted as transmission in per cent [%] (y-axis) against the wavelengths from 900 nm to 4900 nm (x-axis). The membrane proved to be slightly less transparent than the isotropic film, which can be considered as an aspect of IR radiation heatability.

Test V5 (Fig. 8 and Fig. 9)

In test V5, fibre bundles were heat-treated by two IR emitters with a length of 0.2 m_E with different nominal powers, the first IR emitter having a nominal power of 2.1 kW and the second IR emitter having a nominal power of 2.8 kW. The measured values obtained with the first IR emitter are shown as "o" in the graphs; the measured values obtained with the second IR emitter are shown with an "x". The measured permeance (left-hand graph) and selectivity (right-hand graph) are shown normalized to the permeance and selectivity of a fibre bundle that was heat treated with a conventional electric convection oven, thus defining the line at the value y = 1. This can also be referred to as a target value or specification. The measurements were made at powers (x-axis) of the IR emitters from 0.75 kW to 1.6 kW.

The absolute energy input in the heat treatment step at the set power (x-axis) of the respective IR emitter is shown as a numerical value for the respective measured value (symbol). The different energy inputs in kJ are thus derived from the duration of the heat treatment step.

The measurement points shown correspond to the values in Table 2, where column 1 shows the number of individual fibres in the bundle, column 2 shows the nominal power of the IR emitters, column 4 shows the absolute energy input, column 5 shows the normalized O_2 permeance, column 6 shows the normalized O_2/N_2 selectivity, and columns 7 and 8 show the associated standard deviations in relation to the values of columns 5 and 6 respectively.

Table 2: Test results of test V5

Fibres Number [--]	IR Em. Power nom. [kW]	IR Em. Power actual [W]	Energy total [kJ]	Permeance O2 norm. [-]	Selectivity O2/N2 norm. [-]	PD Permeance O2 [-]	PD Selectivity O2/N2 [-]
80	2.1	1550	6.6	1.68	1.03	0.22	0.01
80	2.1	1550	9.7	0.80	0.86	0.00	0.02
80	2.1	960	8.1	1.50	0.79	0.41	0.07
80	2.1	945	11.8	1.11	0.98	0.16	0.06
80	2.8	815	7.1	1.47	0.82	0.31	0.05
80	2.8	825	4.7	1.94	0.80	0.15	0.02
80	2.8	1340	5.9	1.41	1.11	0.50	0.15
80	2.8	1340	8.6	1.35	0.98	0.58	0.17
10	2.1	1550	9.7	0.37	1.01	0.29	0.06
10	2.1	1550	6.6	0.51	1.43	0.08	0.27
10	2.1	955	11.9	1.34	0.77	0.20	0.04
10	2.1	960	8.1	2.06	0.81	0.64	0.27
10	2.8	1340	5.9	0.72	1.22	0.11	0.16
10	2.8	827.5	7.2	1.30	0.81	0.33	0.10
10	2.8	795	4.5	2.00	0.70	0.21	0.09

10	2.8	1300	8.4	0.81	1.05	0.45	0.16

(Key to Table 2: nom.- nominal, SD - standard deviation; IR Em. - IR emitter; norm. – normalized)

The two partial representations I. and II. differ in that, in partial representation I., a fibre bundle of 10 individual fibres was heat treated by the two IR emitters and, in partial representation II., fibre bundles of 80 individual fibres were heat treated in the two IR emitters.

The normalized permeance of 6 indicates complete gas permeability for the gas mixture (left-hand graph), as is the case for a non-heat treated hollow fibre. This value correlates with a normalized selectivity value of 0.4, where there is no separation of the gas mixture into the individual gases O_2 and N_2.

In the graph of Figure 11, the results of Figure 10 for fibre bundles of 10 and 80 individual fibres for the permeance and selectivity are each summarized in a graph. Thus the measuring points of the left-hand graphs I., II. of Figure 10 correspond to those of the left-hand graph of Figure 11. Analogously, the measuring points of the right-hand graphs I., II. of Figure 10 correspond to those of the right-hand graph of Figure 11. The representations of Figure 11 provide for a simple logarithmic plot (y-axis).

For measured values of the fibre bundles composed of 10 individual fibres, the associated measured values are shown in the graphs of Figure 11 as small circles; for the fibre bundles with 80 individual fibres, the associated measured values are shown in the graphs of Figure 11 as "x".

Despite 8 times the number of single fibres, a higher permeance and an only slightly lowered selectivity were observed, based on an exponential, mathematical relationship of the measured values, as indicated by the dashed lines.

To demonstrate the homogeneity of the permeation of the gas over the entire bundle of 80 fibres, the standard deviation of O_2 permeance and O_2/N_2 selectivity was measured by dividing the 80-fibre bundle from heat treatment into 10 partial bundles, which were measured separately with regard to permeation. For permeance, the standard deviation was 25% on average and for selectivity 7%. In view of the large number of experimental sources of error (high proportion of manual activities), the values were an indicator of good homogeneity of the individual fibres of an 80-fibre bundle for heat treatment by means of IR radiation.

All fibre bundles with 80 individual fibres met the (usual) specification for the intended gas separation and were comparable and in some cases better than the fibre bundles heat treated commercially in an electric convection oven.

The eightfold increase in the number of treated individual fibres did not result in a linear relationship with respect to the gas separation properties of the fibre bundle. A correlation of logarithmic permeance and selectivity to the performance of the IR emitters was observed (exponential correlation). Overall, the eight-fold increase in the number of fibres under the experimental conditions resulted in

- a slight decrease in O_2/N_2 selectivity with increasing IR emitter power and
- a 1.3-fold increase in permeance with increasing IR emitter power,

both being plotted and evaluated in the semilogarithmic system.

It turned out surprisingly that with increasing power, the permeance of the fibres decreases faster for fibre bundles of 10 individual fibres (lower dashed line) than for fibre bundles of 80 individual fibres (upper dashed line), as shown in the left-hand graph. Surprisingly, it was also found that with increasing energy input, an almost identical increase in selectivity can be observed for both fibre bundles.

Without being tied to one specific interpretation, the reasons for high, homogeneous separation performance of the hollow fibre heat treated according to the invention are seen as follows:

i) the high transparency (low absorbance) of the fibres in relation to IR radiation, in particular at the wavelength in the maximum power range of the IR emitter (< 5% absorbance (IR absorption)),
ii) refraction of IR radiation and relatively uniform scattering and transmission to adjacent fibres (radiation homogenization), and
iii) high convective energy transport in the heat treatment oven through the gas flow of the gas (convective-thermal homogenization in the heat treatment oven). …

List of reference symbols

100 Plant
102 Polymer solution
104 Process medium
106 Spinning unit
108 Guide roller
109 Alley guide (y direction)

110 Fibre
112 Fibre bundle, partial fibre bundle

120 Coating station (PDMS)

130 Control and regulation unit
132 Data conduit
134 Controller (power)

140 Reserve and supply unit, also provisioning unit
142 Tank
144 Inert gas reservoir
146 Pump (unit)

150 Fixing unit
160 Washing unit
170 Post-treatment unit
180 End-processing unit
182 Fixing station, banding station
184 Cutting station
186 Lifting unit (x-y-z unit)
188 Transport element, transport belt

190 Discharging station
200 Thermal treatment unit
210 Drying station
214 Gas drain conduit
220 Dehumidification station
222 Nozzle unit

224 Gas feed conduit
226 Compressor

230 Fibre accumulator
232 Diversion elements (dynamic, displaceable)

240 Exchange unit

250 Heat treatment station
(thermal treatment station 3)
251 Heat treatment oven
252 IR emitter
254 Reflector
256 Housing
258 Fixation
259 Weight

260 Grouping station

270 Gas feed unit
272 Gas drain (unit)
274 Gas filter unit
280 Conveying means

300 Heating step
302 Ramp-up
304 Power step
306 Cooling step

310 – 328 Characteristic curve
330 Curve of the maxima from 310 – 328

350 Curve for IR absorption
400 Warm-up phase
402 Ramp-up phase
404 Heat treatment phase
406 Cooling phase

A Main conveying direction
B Transmission direction
C Feed conduit
F1, F2 Conveying section

Claims

1. Plant (100) for producing a hollow fibre, in particular a spun hollow fibre, from at least one polymer solution (102), comprising a control and regulation unit (130), a media reserve and supply unit (140), a spinning unit (106) for forming the fibre (110), wherein downstream of the spinning unit (106) for the treatment of the fibre (110) at least the following plant units are included, in the stated sequence:

   a. an exchange unit for solvent exchange (240), in particular comprising a (first) fixing unit (150), in which the fibre directly after the spinning unit (106) can be introduced or passed through, and advantageously comprising at least one washing unit (160) downstream of the fixing unit (150),

   b. at least one aftertreatment unit (170), comprising

   a thermal treatment unit (200) having at least one thermal treatment station,

   characterized in that

   downstream of the exchange unit (240) at least one thermal treatment station of the thermal treatment unit (200) is provided, which is formed as a heat treatment station (250) comprising at least one heat treatment oven (251), and wherein the at least one heat treatment oven (251) comprises at least one infrared emitter (IR emitter) (252), wherein the fibre (110) can be conducted through and/or accommodated at least temporarily in the at least one heat treatment oven (251) comprising the IR emitter (252), and wherein the heat treatment oven (251) is embodied, for the at least temporarily accommodated and/or conductable fibre (110), to heat it for a defined time up to a temperature that is below

the glass softening temperature (T_g), in particular also in the range
i) to 80°C below the glass softening temperature (T_g), and/or
ii) greater than 250°C.

2. Plant according to Claim 1, characterized in that
i) the heat treatment chamber of the heat treatment oven (251) is open to the atmosphere in operation as intended and/or
ii) has at least one gas inlet opening, for regulated (active) introduction of gas into the heat treatment chamber.

3. Plant according to either of the preceding claims, characterized in that the heat treatment oven (251) is embodied, for the at least temporarily accommodated and/or conductable fibre (110), i) to heat it for a defined time up to a limiting temperature of not more than 1°C to 4°C below the glass softening temperature (T_g).

4. Plant according to any of the preceding claims, characterized in that the thermal treatment unit (200) as a further thermal treatment station comprises a drying station (210) upstream of the heat treatment station (250), through which the individual fibres (110) can be conducted as a strand, wherein the drying station (210) comprises
a. - at least one drying element and/or
b. - a feed conduit and a drain conduit for a drying medium, such as in particular a heated gas, and
whereby the interior of the drying station (210) can be heated at least to a temperature of over 30°C, advantageously of over 40°C and ideally of over 50°C, wherein the interior is heatable at maximum to 200°C.

5. Plant according to any of the preceding apparatus claims, characterized in that the thermal treatment unit (200) as a further thermal treatment station comprises a dehumidification station (220) upstream of the heat treatment station (250), in which the individual fibres (110) are conducted through at a distance from one another transversely to the axial direction (longitudinal fibre axis (A_F)), which comprises at least one nozzle element (222) which is directed onto the path of at least one individual fibre (110), and by means of which a gas jet can be guided onto at least one individual fibre (110), in particular a group of individual fibres (110).

6. Plant according to any one of the preceding apparatus claims, characterized in that

i) a grouping station (260) of the grouping unit is arranged ahead of the heat treatment station (250),

ii) the heat treatment station (250) is arranged within a grouping unit, and/or

iii) a grouping unit is arranged between two grouping stations (260), wherein in the grouping station (260) at least one grouping means is provided by means of which at least one group of n individual fibres (110) can be arranged in mutual contact as a bundle (112), wherein n is an integral value from 2 to 180, preferably from 20 to 150, ideally from 30 to 120, wherein optionally up to n = 600 is possible.

7. Plant according to any of the preceding apparatus claims, characterized in that the heat treatment station (250) comprises a ventilation unit or is connected to a ventilation unit and/or the heat treatment oven (251) has at least one gas inlet opening, in particular a gas inlet opening for air.

8. Plant according to any of the preceding apparatus claims, characterized in that the IR emitter (252) has a power relative to the emitter length of

- at least 7.5 kW/m_E, advantageously of at least 10 kW/m_E and ideally of at least 15 kW/m_E and
- of a maximum of 100 kW/m_E, advantageously of a maximum of 50 kW/m_E,

ideally of a maximum of 35 kW/m_E.

9. Plant according to any of the preceding apparatus claims, characterized in that the heat treatment oven (251) comprising the at least one IR emitter (252) has a channel- or alley-like configuration, comprising at least two walls or wall portions parallel to the longitudinal fibre axis (A_F).

10. Plant according to any of the preceding apparatus claims, characterized in that the at least one heat treatment station (250) comprising the heat treatment oven (251) with the IR emitter (252) is arranged at one of the following plant locations:

a. downstream of the washing unit (160), wherein no (further) thermal treatment station of the thermal treatment unit (200) is arranged ahead of the heat treatment station (250),
b. downstream of at least one dehumidification station (220) of a thermal treatment unit (200),
c. downstream of at least one drying station, in particular downstream of a drying station which is arranged downstream of a coating station or application unit.

11. Plant according to any of the preceding apparatus claims, characterized in that downstream of the heat treatment station (250) an end-processing unit (180) is arranged.

12. Plant according to any of the preceding apparatus claims, characterized in that upstream of the heat treatment station (250) a fibre accumulator (230) is arranged.

13. Plant according to any of the preceding apparatus claims, characterized in that it is configured to transport the fibre (110) continuously from the spinning unit (106) to at least the outlet of the heat treatment station (250).

14. Plant according to any of the preceding apparatus claims, characterized in that the post-treatment unit (170) comprises a coating station (120) and/or a discharging station (190).

15. Process for producing a fibre (110) which is spun from a polymer solution (102), in particular for the parallel production of m (individual) fibres (110), comprising the following steps:

- step of spinning the fibre (110) from the polymer solution (102),
- exchange step for the replacement of a solvent, wherein the exchange step may comprise at least one fixing step with a liquid fixing medium and/or at least one washing step in/with a washing medium, in particular in a basin with a washing medium,
- drying step, comprising a heat treatment step, in which the fibre (110) is heated to a temperature below the glass softening temperature (T_g), **characterized in that**

the fibre (110) is heated in the heat treatment step by means of at least one continuously operated IR emitter (252).

16. Process according to Claim 14, characterized in that

during the heat treatment step, the heat treatment of the fibre (110) is accomplished by heating the fibre (110) for a defined time up to a temperature which is below the glass softening temperature (T_g), and

i) is in the range to 80°C below the glass softening temperature (T_g), and/or

ii) is in the range of greater than 250°C.

17. Process according to either of the preceding process claims, characterized in that during the heat treatment step the fibre (110) is heated for a defined time

i) up to a limiting temperature which is not more than 1°C, in particular not more than 4°C, below the glass softening temperature (T_g) and/or

ii) to a limiting temperature in the range of 70°C to 1°C below the glass softening temperature (T_g), and/or

(ii) to a temperature in the range of greater than 280°C to 450°C and below the glass softening temperature (T_g).

18. Process according to any of the preceding process claims, characterized in that during the heat treatment step, the fibre (110) is treated by means of the IR emitter (252) with an IR radiation power based on the emitter length of

- at least 5 kW/m_E, preferably at least 7.5 kW/m_E and ideally at least 10 kW/m_E, and wherein

- the IR radiation power is not more than 100 kW/m_E, advantageously not more than 50 kW/m_E and ideally not more than 35 kW/m_E.

19. Process according to any of the preceding process claims, characterized in that the fibre (110) is produced on a plant (100) which is embodied according to at least one of the preceding apparatus Claims 1 to 14.

20. Process according to any of the preceding process claims, characterized in that the heat treatment step consists at least of two component steps:

- a heating step, in which the IR radiation power is in the range from 5% to 25% of the maximum IR radiation power, advantageously 7.5% to 15%, ideally 10%, and
- a power step, at a maximum IR radiation power of 100 kW/m_E, advantageously of not more than 50 kW/m_E and ideally of not more than 35 kW/m_E.

21. Process according to any of the preceding process claims, characterized in that at least one group of m individual fibres (110) before the heat treatment step is shaped (aligned) in a grouping step to form at least one (partial) bundle (112) of n = 5 to 180 individual fibres (110) and subsequently heat-treated as a bundle (112), wherein either

- n is less than m, so that two or more (partial) bundles (112) are formed, or
- n is equal to m, so that only one bundle (112) is formed.

22. Process according to any of the preceding process claims, characterized in that the fibre (110) is transported continuously from the spinning unit (106) to at least the outlet of the heat treatment station (250).

23. Process according to any of the preceding process claims, characterized in that the heat treatment time is in the range of 0.1 to 60 s, preferably 0.5 to 30 s, ideally 1 to 20 s.

24. Process according to any of the preceding process claims, characterized in that the heat treatment oven

i) is open to the atmosphere, in particular has an inlet and/or outlet opening for the fibre and a

passive air entry takes place with the conveyed fibre into the heat treatment oven and/or

ii) comprises or is connectable to a ventilation station, via which a gas mixture, in particular, air, N_2 and/or CO_2, is introduced into the interior of the heat treatment oven.