precipitating bath, and process using the same

bain de précipitation, et procédé utilisant ce bain de précipitation

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Description

Field of the invention

[0001] The present invention relates to a precipitating bath for holding a coagulation liquid for the precipitation of shaped bodies formed from a spinning solution, the bath having a bottom and at least one precipitating bath discharge nozzle mounted in the bottom, the at least one discharge nozzle having an intake port and opposite it a discharge port for the passage therethrough of the shaped bodies formed from the spinning solution, the bath also having at least one inlet for the coagulation liquid and at least one outlet for the coagulation liquid. Furthermore, the present invention relates to a process for the preparation of cellulotic shaped bodies, in which process a solution of cellulose in a tertiary amine N-oxide and, optionally, water is shaped in the hot state and the shaped solution is extruded into a gaseous medium over a predefined gas zone (gap), and the extruded shaped solution is then introduced into a coagulation liquid for precipitation.

Background art

[0002] Such a precipitating bath and such a process are known, for example from JP-A-61-19805. In that publication a device for high-speed wet spinning is disclosed, where the starting spinning solution is spun from a nozzle into a spinning funnel. Inside this funnel the spinning solution comes into contact with the coagulation liquid, which is conveyed along the wall of the spinning funnel, causing precipitation of the shaped bodies. Inside the funnel a great speeding up of the spinning solution takes place. As a result of this, however, there may be turbulence at the point of impact of the spinning solution onto the coagulation liquid. This turbulence often results in spinning ruptures, so that the use of the precipitation bath described in JP-A-61-19805 and of the process also described there produces a stable spinning process for only a very few cellulotic spinning solutions. Admittedly, in the examples of JP-A-61-19805 winding rates of up to 1,500 m/min are mentioned. However, this is attained by a complex and expensive arrangement of several such acceleration funnels, as a result of which the starting up (stringing up) on the one hand and a stable spinning process on the other hand are made considerably more difficult.

[0003] A process for the preparation of cellulotic fibres by extruding a spinning solution containing dissolved cellulose into a gaseous medium over a predefined gas zone (gap), followed by its immersion in and conveyance through a coagulation bath over a predetermined coagulation zone, the drawing of the obtained cellulotic fibres and their winding up is also known from EP-A-0,817,873. In this process the extruded spinning solution after the gas zone is accelerated to a defined speed and introduced into a coagulation bath flowing laminarly in at least about the approximate direction of the extruded spinning solution. The coagulation liquid in that case is fed laterally to the path of the spinning solution, and the directions of flow of the spinning solution and of the coagulation liquid are kept approximately parallel over the entire coagulation zone, and the resulting cellulose fibres on leaving the coagulation zone are deflected laterally and then wound up.

Disclosure of the invention

[0004] It is true that the process disclosed in EP-A-0,817,873 reduces the drawbacks of JP-A-61-19805 which are caused by the turbulence in the spinning funnel, and it also guarantees a more stable process. Furthermore, the complexity and cost of the apparatus for carrying out the process described in EP-A-0,817,873 are considerably lower. However, at high spinning rates and a simultaneous high throughput the method disclosed in EP-A-0,817,873 may also lead to instabilities in the process and thus to non-uniformity in the obtained yarns or even to spinning ruptures. These instabilities are due, amongst other things, to the fact that in the precipitation of shaped bodies formed from a spinning solution using the devices and/or processes known from the prior art there is often conglutination of these shaped bodies.

[0005] The present invention seeks first of all to provide a device for the precipitation of shaped bodies formed from a spinning solution which at least reduces the prior art drawbacks described above. A further object of this invention is to make available a process with the aid of which there can be conglutination-free and stable spinning even at high throughputs and/or spinning rates.

[0006] The first of these problems is surprisingly solved by the provision of a precipitating bath of the type described in the opening paragraph, characterised in that it has a cover sheet with at least one hole for holding the at least one precipitating bath discharge nozzle, the cover sheet being positioned above the at least one inlet for the coagulation liquid and beneath the at least one outlet for the coagulation liquid, and that in the at least one precipitating bath discharge nozzle in the at least one hole of the cover sheet is arranged such that one or more orifices are formed in the cover sheet in a ring around the at least one precipitating bath discharge nozzle.

[0007] Because there are generally a plurality of, frequently many, precipitating bath discharge nozzles present in a precipitating bath and consequently the cover sheet generally contains a plurality of, frequently many, holes for holding the precipitating bath discharge nozzles, the term "precipitating bath discharge nozzles" in the following description also encompasses "a precipitating bath discharge nozzle" and vice versa. The skilled person will recognise that the function of the precipitating bath within the framework of the present invention is not characterised and/or limited by the number of precipitating bath discharge nozzles present in it nor by the number
of holes in the cover sheet associated with these nozzles. The precipitating bath according to the invention is especially useful in those cases where the spinning solution is a solution of cellulose in a tertiary amine N-oxide, in particular N-methylmorpholine N-oxide, and, optionally, water. As is known, in such cases the cellulose prior to being spun is dissolved at elevated temperature, generally at about 55 to 130°C in an appropriate solvent, such as a tertiary amine N-oxide and, optionally, water. The solution of the cellulose in the tertiary amine N-oxide and, optionally, water is extruded in the hot state with the aid of a spinneret and is formed (shaped) in that operation. The thus shaped solution is passed to a coagulation bath generally filled with substances which do not dissolve cellulose, for example an aqueous solution, in which procedure the tertiary amine N-oxide is extracted from the shaped solution and the cellulose precipitates in the shaped state. Between the spinneret and the coagulation bath cooling of the shaped solution generally takes place in a so-called air gap. This cooling can be carried out with ambient gas, for example air, alone, but in general the cooling is carried out with the aid of additional blown-in gas, for example with conditioned air.

According to the invention, the precipitating bath has a so-called cover sheet, which is constructed in such a way that one or more holes in it coincide with the one or more precipitating bath discharge nozzles present, as a result of which orifices are formed in a ring around the precipitating bath discharge nozzle(s). The cover sheet may be of sheet metal. The precipitating bath discharge nozzle in that case is generally mounted in such a way that only its upper part, i.e. its intake port, is near the ring of orifices formed by these nozzles and the cover sheet. It is preferred for the cover sheet to be positioned such that it forms a plane with the upper edge of the precipitating bath discharge nozzle, i.e. terminates flush with it, and none of the parts of the precipitating bath discharge nozzle extend above the plane formed by the cover sheet.

Thus, according to the invention, one or more orifices are formed in the cover sheet in a ring around the at least one precipitating bath discharge nozzle. This can easily be done in such a way that the cover sheet contains for instance circular holes, the diameter of which in each case exceeds the diameter of the precipitating bath discharge nozzles arranged in them. By means of such a construction, orifices, for example ring-shaped slits, are formed or remain in the cover sheet around the precipitating bath discharge nozzle. It is also possible, although not preferred, for the holes in the cover sheet to surround the precipitating bath discharge nozzles tightly while at the same time in addition other, for example smaller, holes are arranged as orifices in a ring in the cover sheet around the orifice which is now closed off by the precipitating bath discharge nozzle, i.e. that the cover sheet has a perforation at this point.

The orifices formed in the cover sheet in a ring around the precipitating bath discharge nozzle can be continuous or discontinuous. The ring-shaped slot around the precipitating bath discharge nozzle already described above serves as an example of a continuous orifice. However, it is more preferred for the orifices to have a discontinuous form. In an advantageous embodiment the precipitating bath has a cover sheet of which the hole or holes have at least one land leading inward from the edge of this hole or these holes in the direction of the centre of the hole in question. When such a hole is arranged around the precipitating bath discharge nozzle, then an orifice is formed in a circle around the nozzle and is interrupted by the land.

More advantageously, the hole or holes of the cover sheet in each case have six lands leading inward from the edge of the hole in the direction of the centre of the hole, which respective lands are staggered by 60° in relation to one another. In the case of circular holes these lands will form a wreath-like structure which, for instance when all lands are of equal length, which is preferred, again forms an "inner" hole with a smaller diameter. When the precipitating bath discharge nozzle is mounted inside this wreath in such a way that it is at the centre of this wreath, i.e. tightly adjacent to all the lands, then an orifice of in each case six annular ring-shaped slits is formed through the cover sheet around the precipitating bath discharge nozzle.

The cover sheet and the precipitating bath discharge nozzle are arranged such that they are immersed in the coagulation liquid during operation of the precipitating bath. This means that the spinning solution first covers a predetermined distance through the coagulation liquid before it comes upon the intake port of the precipitating bath discharge nozzle. The discharge for the coagulation liquid is positioned above the cover sheet. The coagulation liquid thus leaves the precipitating bath essentially above the cover sheet. Of course, a portion of the coagulant is also discharged via the precipitating bath discharge nozzle. The level of the coagulation liquid in the case of the precipitating bath according to the invention is preferably adjusted at the discharge point with at least one overflow weir (spillway) to regulate the filling height of the coagulation liquid. In a suitable embodiment the precipitating bath has for instance a rectangular basic shape with four side walls, of which two side walls opposite to one another, henceforth called the longitudinal sides, are longer than the other two sides opposite one another. If for instance the side walls of the two longitudinal sides are somewhat lower than the other two side walls, then the discharge takes place by way of the lower side walls, and the height of the walls of the longitudinal sides determines the level of the coagulation liquid, i.e. the longitudinal sides act as overflow weirs. Of course the quantities of coagulation liquid which overflow can be collected and re-used. This may be done, for example, by means of suitable overflow channels and recirculation of the coagulation liquid back into the precipitating bath. In that case of course it may need to be ensured that the predetermined composition of the coagulation liquid, of-
ten a mixture of water and N-methyl-morpholine N-oxide (NMMO), is maintained. Optionally, one or more components of the recirculated material should be enriched or depleted prior to being returned.

[0014] It is preferred for the height of the overflow weirs to be adjustable. This can be done, for example, by exchanging the corresponding side walls. The height of the overflow weirs and hence of the level of the coagulating liquid depends in the first place on the cellulosic shaped bodies to be formed and the spinning conditions. Thus, in the production of yarns or multifilaments linear density (titer) and spinning rate, for example, have an effect.

[0015] In a further special embodiment of the precipitating bath the overflow weirs have a saw-tooth profile. As a result of this an even more uniform discharge of the coagulant over the entire length of the precipitating bath can be achieved.

[0016] According to the invention, the inlet for the coagulation liquid is located beneath the cover sheet. As a result of the inlet for the coagulation liquid being located beneath the cover sheet and the positioning of the outlet above the cover sheet, the position of which can be set with the aid of the overflow weirs, a flow stream of the coagulation liquid from the bottom to the top is set up inside the precipitating bath. In this process the stream of coagulation liquid leaves through the orifices in the cover sheet arranged in a ring around the precipitating bath nozzle, for example through the formed ring-shaped slits. Since the shaped bodies to be precipitated encounter the coagulation liquid from above and then cover a predetermined distance inside the coagulation liquid before they come upon the intake port of the precipitating bath nozzle, the coagulation liquid thus flows over a predetermined distance towards the shaped bodies to be precipitated. In other words, the precipitating bath according to the invention is a countercurrent precipitating bath, where the coagulation liquid has a direction of flow opposite to the direction of travel of the entering shaped bodies, for example turned through about 180° calculated on this direction of travel, at least at or near the entry of the shaped bodies into the precipitating bath discharge nozzle. The countercurrent flow leads to a clear improvement as regards the precipitation and the quality of the precipitated shaped bodies. It is assumed that, because of this countercurrent flow, the formation of a vortex (spout) in the coagulation liquid at the point of entry of the shaped bodies to be precipitated into the precipitation bath is avoided or reduced, which prevents conglutination inside the shaped bodies, for example among the monofilaments of a thread. This positive effect becomes especially noticeable when spinning filaments, preferably multifilament threads of high linear density (titer), at high rates, such as 100 to 1,500 m/min, particularly 500 to 1,000 m/min, more particularly 800 to 1,200 m/min. For that reason the precipitating bath according to the present invention is especially suitable for use in filament spinning at high rates.

[0017] The supply of the coagulation liquid to the precipitating bath preferably takes place via distributing pipes, which are situated beneath the cover sheet and for example run parallel to the longitudinal walls of the precipitating bath. These distributing pipes are equipped with several small exit holes over the entire length of the pipes, through which the coagulation liquid leaves. This leads to a uniform discharge of the coagulation liquid. It is advantageous for the distributing pipes to extend over the entire length of the precipitating bath and the exit holes to be distributed evenly over the length of the pipes.

[0018] In a preferred embodiment the distributing pipes are arranged in such a way that the exit holes point slantingly downwards, for example at an angle of 45°, in the direction of the bottom of the precipitating bath. This produces an even more effective flow movement in the precipitating bath according to the invention. The indirect supply of the coagulating liquid to the precipitating bath via the distributing pipes over the entire length of the precipitating bath moreover ensures a uniform distribution of the coagulation solution with regard to temperature and additives, for instance NMMO, over the entire length of the precipitating bath. Furthermore, possible intermingling of the yarns is counteracted. It is further preferred that the coagulation liquid be supplied to the precipitating bath discharge nozzle from two directions, for instance symmetrically. This can be effected, for example, by means of two distributing pipes each running parallel to the longitudinal sides of the precipitating bath and parallel to a row of precipitating bath discharge nozzles. This produces an even better uniformity of the supply of coagulation liquid and prevention of turbulences.

[0019] The precipitating bath discharge nozzles of the precipitating bath according to the present invention may be connected to the bottom of the precipitating bath, preferably removably so. The intake port is located within the coagulation liquid, and the discharge ports are embedded in the bottom and thus make possible the discharge of the precipitated shaped bodies, e.g. filaments, from the precipitating bath and their subsequent take-up, for example by means of winding. Of course, together with the precipitated shaped bodies exiting from the precipitating bath some coagulation liquid is also dragged out, for instance because it adheres to the filaments which are leaving. To regenerate this coagulation liquid and/or to make the drying process easier, it may be desirable to separate the shaped bodies from the coagulation liquid adhering to them. This can be done, for example, by a slantwise withdrawal of the shaped bodies from the precipitating bath discharge nozzle. This discharge enables the adhering liquid to be stripped off and conveyed back to the precipitating bath.

[0020] The precipitating bath discharge nozzles may be made of an inert material, such as a metal or ceramic material. It has been shown that the stripping off of adhering liquid from the shaped bodies can be performed especially advantageously and gently for the resulting precipitated shaped bodies if the precipitating bath discharge nozzle of the precipitating bath according to the
invention is made of high-grade (stainless) steel which has subsequently been subjected to matt chromium plating, which results in a so-called "orange peel" structure.

The bottom of the precipitating bath according to the present invention can have an uneven profile. For example, when the precipitating bath discharge nozzles are arranged in a row in the middle of the bottom of the precipitating bath running parallel to the longitudinal sides of the precipitating bath, it can be an advantage if this central area of the bottom is positioned higher, i.e. closer towards the cover sheet, than the areas of the bottom on either side. In these latter two areas the distributing pipes can in that case conveniently be arranged. Such an arrangement is especially preferred because as a result the depth of the bath, i.e. the depth of the coagulation liquid through which the shaped bodies formed from the spinning solution are passed, can be kept low. Especially preferably, the depth of the coagulation liquid at the point beneath the precipitating bath discharge nozzle is only about 10 to 40 mm, more preferably still about 10 to 20 mm. Surprisingly, it has been found that this low depth of the precipitating bath at any rate significantly reduces the formation of vortexes when passing the shaped bodies through the coagulation liquid. As a result, the shaped bodies are brought into significantly better contact with the coagulation liquid, which leads to a significant improvement of the filling. Because of this smaller depth of the precipitating bath there are also lower frictional losses at high spinning rates.

Furthermore, it is preferred if the cover sheet of the precipitating bath according to the invention contains still further holes (at least one), which are connected to the bottom of the precipitating bath by means of a small-diameter (capillary) tube. These further holes in the cover sheet serve as so-called "emergency exits". These emergency exit holes in the cover sheet are connected to capillary tubes fitted into the precipitating bath, the top edges of which terminate flush with the cover sheet, without further orifices being formed in the process. These capillary tubes enable a free passage right through the bottom of the precipitating bath into the open. The additional holes in the cover sheet together with the capillary tubes connected thereto are useful at the start of the precipitation, for example when stringing up, in order to be able in a first step to pull the individual threads through the whole precipitating bath as rapidly and smoothly as possible. After that, the threads are separated and threaded into the respective precipitating bath discharge nozzles. If during the operation of the spinneret one or several spinning positions do not run stably, then the corresponding threads or also monofilaments of a thread can be discharged via these emergency exits. In this way clogging of the precipitating bath discharge nozzles is avoided. After the stringing up or in the case of non-use the capillary tubes can be sealed, for example from below with conical rubber plugs.

The present invention also provides a process for the preparation of cellulosic shaped bodies, in which process a solution of cellulose in a tertiary amine N-oxide and, optionally, water is shaped in the hot state and the shaped solution is extruded into a gaseous medium over a predefined gas zone (gap), after which the extruded shaped solution is introduced into a coagulation liquid for precipitation, characterised in that during the precipitation the coagulation liquid flows in countercurrent to the direction of movement of the shaped solution, at least in the region of the inlet of the shaped solution into the coagulation liquid.

The flow of the coagulation liquid during precipitation in a direction counter to the direction of movement of the shaped cellulose solution to be precipitated leads to a uniform and effective precipitation, which has a favourable effect on the properties of the shaped bodies obtained. This is especially the case with regard to the thread (fibre) properties when spinning cellulosic fibres and filaments using this countercurrent process.

It is preferred in this case if the claimed process for the precipitation of cellulosic shaped bodies is performed using a precipitating bath according to the invention.

When the cellulosic shaped bodies made by the process according to the invention are cellulosic fibres or filaments, then advantageously on leaving the precipitating bath these fibres or filaments are subsequently treated, as in a manner known per se, for example by washing, applying a finish, drying and winding up.

It is especially preferred in that case if the cellulosic fibres or filaments on leaving the precipitating bath, such as a precipitating bath according to the invention, are first diverted laterally in contact with an edge of the discharge port of the at least one precipitating bath discharge nozzle and then treated further. It is still more preferred if the lateral diversion of the cellulosic fibres or filaments is carried out in such a way that they run over one edge of the discharge port of the one or more precipitating bath discharge nozzles. In this way good removal of the adhering and entrained coagulation liquid is achieved.

Overall, the process according to the invention makes a uniform precipitation process (coagulation) for all threads possible. The resulting threads have high uniformity with regard to their physical, optical and textile properties. The process according to the invention in particular enables the spinning of filaments, preferably multifilament threads of high linear density, at high rates of from 100 to 1,500 m/min, particularly from 500 to 1,000 m/min, more particularly from 800 to 1,200 m/min.

A special benefit of the precipitating bath according to the invention and the process according to the present invention lies in the fact that, in the spinning of filaments, products can be obtained which stand out because of their excellent uniformity in dye uptake (absorption). In fabrics, particularly woven fabrics, made of such filaments this effect becomes noticeable by way of a significant reduction in streakiness and tendency to streak. This positive result is attributed to the special geometry
of the precipitating bath, the low depth of the precipitating bath at the point of the precipitating bath discharge nozzle being especially worth mentioning. It came as a complete surprise that this low depth of the precipitating bath apparently leads decisively to the described advantages, an effect which could in no way be foreseen.

**Description of the drawings**

[0030] The invention will be further explained with reference to the accompanying drawings by way of example. In the drawings Figure 1 is a schematic figure which shows the cross-section of a suitable embodiment of the precipitating bath according to the invention. Figure 2 is a schematic figure which shows an overflow weir with a saw-tooth profile in side view, and Figure 3 is a schematic figure which shows a cover sheet in plan view from above.

[0032] The precipitating bath is positioned beneath a spinneret 1 and an air gap 2. The precipitating bath can be adjusted in height, i.e. in the length of the distance between the spinneret 1 and the precipitating bath, with the aid of an adjusting screw. By this means the length of the air gap 2, i.e. the distance between the spinneret 1 and the precipitating bath, is adjusted.

[0033] The precipitating bath shown as an example in Fig. 1 has a length of about 1,000 mm and a width of about 250 mm. At the two long sides of the bath is found in each case an exchangeable overflow weir 5 with a saw-tooth profile and an overflow channel 12. At one narrow side of the bath there is the connection to an inlet supply line for the coagulation liquid 4. The supply line lies in the middle and, with the aid of a T-piece and two inserted distributing pipes 8 sealed via an O-ring, liquid is distributed over the two long sides of the precipitating bath. The two distributing pipes 8 have an outer diameter of 20 mm and run parallel to the long sides of the precipitating bath. They each have 40 exit holes, in order to distribute the coagulation liquid 4 uniformly over the entire length of the precipitating bath. The distributing pipes 8 are inserted in such a way that the exit holes point downwards at an angle of 45° to the horizontal. The back ends, i.e. the ends opposite to the insertion side at the T-piece, of the distributing pipes 8 are closed off with stoppers, sealing taking place with polytetrafluoroethylene tape. In the middle of the precipitating bath lie precipitating bath discharge nozzles 10, which are arranged parallel to the long sides of the bath. Each fibre (thread) 11, composed of multiple monofilaments 3, from the spinneret 1 passes through a precipitating bath discharge nozzle 10. The precipitating bath discharge nozzles 10 are removable and when used in the bath are retained by O-rings and simultaneously sealed off in the direction of the bath. The precipitating bath discharge nozzles 10 made of stainless steel, which has been subsequently matt chromium plated. The matt chromium plating gives an "orange peel" structure to the surface.

[0034] In the precipitating bath lies a cover sheet 6 with holes into which the precipitating bath discharge nozzles 10 are fitted. The cover sheet terminates flush with the top edges of the precipitating bath discharge nozzles 10. In addition there are six further holes in the cover sheet, the so-called emergency exit holes.

[0035] The emergency exit holes in the cover sheet 6 are connected to capillary tubes 7, which have been fitted into the precipitating bath and the top edges of which terminate flush with the cover sheet 6. The capillary tubes 7 run parallel to the precipitating bath discharge nozzles. Down below, i.e. at the bottom of the precipitating bath, there are holes in the bath, making it possible for the capillary tubes 7 to pass through freely. When they are not needed, these emergency exit capillary tubes 7 can be closed up with conical rubber stoppers 9. The emergency exit capillary tubes 7 have been welded into the precipitating bath at the bottom.

[0036] Fig. 2 shows the overflow weir 5 of Figure 1 in a schematic side view. The saw-tooth profile, which enables a very uniform overflow of the coagulant to be set, is clearly recognisable.

[0037] Figure 3 shows the cover sheet 6 of Figure 1 in top plan view. Recognisable in this view are first of all holes 13 for the precipitating bath discharge nozzles 10. Each hole 13 additionally shows six lands 14 staggered by 60° in relation to one another. These lands point inward, which results in a defined inner diameter of the holes 13. The precipitating bath discharge nozzles 10 fit exactly into this inner diameter of the holes 13. Further, the holes 13 for the precipitating bath discharge nozzles 10. The precipitating bath discharge nozzles 10 fit exactly into this inner diameter of the holes 13. Further, Figure 3 shows emergency exit holes 15 for the emergency exit capillaries 7 represented in Figure 1.

[0038] The top edge of these emergency exit holes 15 has been counterbored at an angle of 45°, in order to avoid a sharp edge. The distribution of the two different hole types 13 and 15 on the cover sheet 6 takes place in such a way that there is always an emergency exit hole 15 midway between two holes 13 for the precipitating bath discharge nozzles 10, i.e. an emergency exit is always provided for two fibres 11.

[0039] The precipitating bath shown in Figure 1 is a countercurrent precipitating bath, i.e. the coagulation liquid 4 has a direction of flow, indicated in Figure 1 by direction arrows, that is at 180° relative to the flow direction of the fibres or monofilaments entering this coagulation liquid.

[0040] The coagulation liquid is circulated via a container (reservoir). This circulation consists of a container, a concentration controller for the constituents of the coagulation liquid 4, a pump, a heat exchanger with temperature control, a filtration system, a flow meter for setting the desired volume stream, and the precipitating bath. The excess coagulation liquid, consisting of the coagulation liquid overflow 12 of the precipitating bath and the stripped off coagulation liquid 4 from the fibres 11, runs back into the container. The precipitating bath circulation is run at a constant temperature, with a temperature of 5 to 30°C being set. Since the wet fibres 11 on leaving the precipitating bath also continually drag out...
some coagulation liquid 4 which is not stripped off, this loss in the precipitating bath circulation has to be replaced. Furthermore, as a result of diffusion of, for example, NMMO from the filaments into the coagulation liquid, a build up of NMMO is formed in the coagulation liquid. The replenishment of any of the precipitating bath components, such as water, to compensate for the losses due to dragging out and to maintain the desired NMMO concentration takes place by way of an NMMO concentration controller. Of course, it is also possible to add further additives in this way, for instance to avoid accumulation or sedimentation or to change the properties of the shaped bodies.

[0041] As was described earlier, in operation the individual filaments 3 of each fibre 11 enter the coagulation liquid 4 from the top towards the bottom and are withdrawn through the precipitating bath discharge nozzle 10 aslant downwards - in order to strip off the coagulation liquid 4 from fibres 11. In addition, the precipitating bath surface is stabilised by means of the cover sheet 6 placed in the precipitating bath, i.e. a uniform, constant precipitating bath height is thus guaranteed over the entire precipitating bath surface, and no turbulent streams arise.

Claims

1. A precipitating bath for holding a coagulation liquid for the precipitation of shaped bodies formed from a spinning solution, the bath having a bottom and at least one precipitating bath discharge nozzle mounted in the bottom, the at least one discharge nozzle having an intake port and opposite it a discharge port for the passage therethrough of the shaped bodies formed from the spinning solution, the bath also having at least one inlet for the coagulation liquid and at least one outlet for the coagulation liquid, characterised in that the precipitating bath has a cover sheet with at least one hole for holding the at least one precipitating bath discharge nozzle, the cover sheet being positioned above the at least one inlet for the coagulation liquid and beneath the at least one outlet for the coagulation liquid, and in that the at least one precipitating bath discharge nozzle in the at least one hole in the cover sheet is arranged such that one or more orifices are formed in the cover sheet in a ring around the at least one precipitating bath discharge nozzle.

2. A precipitating bath according to claim 1, characterised in that the spinning solution is a solution of cellulose in a tertiary amine N-oxide, preferably N-methylmorpholine N-oxide, and, optionally, water.

3. A precipitating bath according to claim 1 or 2, characterised in that the at least one precipitating bath discharge nozzle in the at least one hole of the cover sheet is arranged such that the intake port of the at least one precipitating bath discharge nozzle is flush within the plane of the cover sheet.

4. A precipitating bath according to any of claims 1 to 3, characterised in that the at least one hole of the cover sheet has at least one land leading inward from the edge of this hole towards the centre of the hole.

5. A precipitating bath according to claim 4, characterised in that the at least one hole of the cover sheet has six lands leading inward from the edge of the hole towards the centre of the hole, the lands being staggered by 60° in relation to one another.

6. A precipitating bath according to any of claims 1 to 5, characterised in that the discharge for the coagulation liquid from the precipitating bath contains at least one overflow weir for controlling the filling height of the coagulation liquid,

7. A precipitating bath according to claim 6, characterised in that the at least one overflow weir has a saw-tooth profile.

8. A precipitating bath according to any of claims 1 to 7, characterised in that the at least one precipitating bath discharge nozzle is made of stainless steel and has subsequently been subjected to a matt chromium plating.

9. A precipitating bath according to any of claims 1 to 8, characterised in that the cover sheet has at least one further hole, which is connected to the bottom of the precipitating bath by means of a capillary tube.

10. The use of a precipitating bath according to any of claims 1 to 9 for spinning multifilament fibres at rates from 100 to 1,500 m/min, preferably from 800 to 1,200 m/min.

11. A process for the preparation of cellulosic shaped bodies, in which process a solution of cellulose in a tertiary amine N-oxide and, optionally, water is shaped in the hot state and the shaped solution is extruded into a gaseous medium over a predefined gas zone, after which the extruded shaped solution is introduced into a coagulation liquid for precipitation, characterised in that during the precipitation the coagulation liquid flows in countercurrent to the direction of movement of the shaped solution, at least in the region of the inlet of the shaped solution into the coagulation liquid.

12. A process according to claim 11, characterised in that the precipitation of the cellulosic shaped bodies into a precipitating bath is carried out in a precipitating bath according to any of claims 1 to 9.
13. A process according to claim 12, characterised in that the cellulose shaped bodies are cellulose fibres or filaments, which are further treated after leaving the precipitating bath.

14. A process according to claim 13, characterised in that the cellulose fibres or filaments leaving the precipitating bath are first diverted laterally in contact with an edge of the discharge port of the at least one precipitating bath discharge nozzle and are then treated further.

15. A process according to any of claims 11 to 14, characterised in that the multifilament threads are spun at rates of from 100 to 1,500 m/min, preferably from 800 to 1,200 m/min.

**Patentansprüche**

1. Ausfällungsbad für das Halten einer Koagulationsflüssigkeit für das Ausfällen geformter Körper, die aus eines Spinnglück lösung geformt werden, wobei das Bad einen Boden und mindestens eine Ausfalls- bauslussdüse aufweist, die im Boden montiert ist, wobei die mindestens eine Auslassdüse eine Einlassöffnung und eine gegenüberliegende Auslassöffnung für das Hindurchgehen der geformten Körper, die aus der Spinnlösung gebildet werden, aufweist, wobei das Bad ebenfalls mindestens einen Einlass für die Koagulationsflüssigkeit und mindestens einen Auslass für die Koagulationsflüssigkeit aufweist, dadurch gekennzeichnet, dass das Ausfällungsbad eine Deckplatte aufweist mit mindestens einem Loch für das Halten der mindestens einen Ausfallsbadauslassdüse, wobei die Deckplatte über dem mindestens einen Einlass der Koagulationsflüssigkeit und unter dem mindesten einen Auslass für die Koagulationsflüssigkeit positioniert ist, und derart, dass die mindestens eine Ausfallsbadauslassdüse in dem mindestens einen Loch in der Deckplatte so angeordnet ist, dass eine oder mehrere Öffnungen in der Deckplatte in einem Ring um die mindestens eine Ausfallsbadauslassdüse gebildet wird bzw. werden.


3. Ausfällungsbad nach Anspruch 1 oder 2, dadurch gekennzeichnet, dass die mindestens eine Ausfallsbadauslassdüse in dem mindestens einen Loch der Deckplatte so angeordnet ist, dass die Einlassöffnung der mindestens einen Ausfallsbadauslassdüse mit der Ebene der Deckplatte bündig ist.

4. Ausfällungsbad nach einem der Ansprüche 1 bis 3, dadurch gekennzeichnet, dass das mindestens eine Loch der Deckplatte mindestens eine Anschlussfläche aufweist, die nach innen von der Kante dieses Lochs auf den Mittelpunkt des Lochs zu führt.

5. Ausfällungsbad nach Anspruch 4, dadurch gekennzeichnet, dass das mindestens eine Loch der Deckplatte sechs Anschlussflächen aufweist, die nach innen von der Kante des Lochs auf dem Mittelpunkt des Lochs zu führen, wobei die Anschlussflächen mit Bezug aufeinander um 60 ° versetzt sind.

6. Ausfällungsbad nach einem der Ansprüche 1 bis 5, dadurch gekennzeichnet, dass der Auslass für die Koagulationsflüssigkeit aus dem Ausfällungsbad mindestens ein Überlaufwehr zum Regulieren der Füllhöhe der Koagulationsflüssigkeit enthält.

7. Ausfällungsbad nach Anspruch 6, dadurch gekennzeichnet, dass das mindestens eine Überlaufwehr ein Sägezahnprofil aufweist.

8. Ausfällungsbad nach einem der Ansprüche 1 bis 7, dadurch gekennzeichnet, dass die mindestens eine Ausfallsbadauslassdüse aus Edelstahl hergestellt ist und daraufhin einer Mattchromplattierung unterzogen worden ist.

9. Ausfällungsbad nach einem der Ansprüche 1 bis 8, dadurch gekennzeichnet, dass die Deckplatte mindestens ein weiters Loch aufweist, das durch eine Kapillarröhre an den Boden des Ausfällungsbads angeschlossen ist.

10. Verwendung eines Ausfällungsbads nach einem der Ansprüche 1 bis 9 zum Spinnen von Multifilamentfasern mit Geschwindigkeiten von 100 bis 1.500 m/min, bevorzugt 800 bis 1.200 m/min.


12. Verfahren nach Anspruch 11, dadurch gekennzeichnet, dass die Ausfällung des geformten Cel-
Bain de précipitation destiné à recevoir un liquide de coagulation, caractérisé en ce que la feuille de recouvrement est agencée de telle sorte qu’un orifice d’entrée dudit au moins un trou de la feuille de recouvrement est aligné sur le plan de la feuille de recouvrement.

Revendications

1. Bain de précipitation destiné à recevoir un liquide de coagulation en vue de la précipitation de corps façonnés formés à partir d’une solution de filage, le bain présentant un fond et au moins un ajutage de décharge monté dans le fond du bain de précipitation, ledit au moins un ajutage de décharge présentant un orifice d’entrée et face à celui-ci un orifice de décharge qui permet le passage des corps façonnés formés à partir de la solution de filage, le bain présentant également au moins une entrée pour le liquide de coagulation et au moins une sortie pour le liquide de coagulation, caractérisé en ce que le bain de précipitation présente une feuille de recouvrement dotée d’au moins un trou et qui retient ledit au moins un ajutage de décharge du bain de précipitation, la feuille de recouvrement étant placée au-dessus de ladite au moins une entrée de liquide de coagulation et en dessous de ladite au moins une sortie de liquide de coagulation, et en ce que ledit au moins un ajutage de décharge du bain de précipitation situé dans ledit au moins un trou de la feuille de recouvrement est agencé de telle sorte qu’un ou plusieurs orifices sont formés dans la feuille de recouvrement en un anneau qui entoure ledit au moins un ajutage de décharge du bain de précipitation.

2. Bain de précipitation selon la revendication 1, caractérisé en ce que la solution de filage est une solution de cellulose dans un N-oxide d’amine tertiaire, de préférence un N-oxide de N-méthylmorpholine, et facultativement d’eau.

3. Bain de précipitation selon les revendications 1 ou 2, caractérisé en ce que ledit au moins un ajutage de décharge du bain de précipitation prévu dans ledit au moins un trou de la feuille de recouvrement est agencé de telle sorte que l’orifice d’entrée dudit au moins un ajutage de décharge du bain de précipitation soit aligné sur le plan de la feuille de recouvrement.

4. Bain de précipitation selon l’une quelconque des revendications 1 à 3, caractérisé en ce que ledit au moins un trou ménagé dans la feuille de recouvrement présente au moins une surface orientée vers l’intérieur depuis le bord de ce trou et en direction du centre du trou.

5. Bain de précipitation selon la revendication 4, caractérisé en ce que ledit au moins un trou ménagé dans la feuille de recouvrement présente six surfaces orientées vers l’intérieur depuis le bord du trou et en direction du centre du trou, les surfaces étant décalées de 60° les unes par rapport aux autres.

6. Bain de précipitation selon l’une quelconque des revendications 1 à 5, caractérisé en ce que la décharge du liquide de coagulation hors du bain de précipitation contient au moins une barrière à surverse qui contrôle la hauteur de remplissage du liquide de coagulation.

7. Bain de précipitation selon la revendication 6, caractérisé en ce que ladite au moins une barrière à surverse a un profil en dents de scie.

8. Bain de précipitation selon l’une quelconque des revendications 1 à 7, caractérisé en ce que ledit au moins un ajutage de décharge du bain de précipitation est réalisé en acier inoxydable qui a ensuite subi un plaquage mat au chrome.

9. Bain de précipitation selon l’une quelconque des revendications 1 à 8, caractérisé en ce que la feuille de recouvrement présente au moins un autre trou qui est relié au fond du bain de précipitation par un tube capillaire.

10. Utilisation d’un bain de précipitation selon l’une quelconque des revendications 1 à 9, pour le filage de fibres multifilamentaires à des débits de 100 à 1 500 m/min et de préférence de 800 à 1 200 m/min.

11. Procédé de préparation de corps façonnés en cellulose, dans lequel une solution de cellulose dans un N-oxide d’amine tertiaire et facultativement d’eau est façonnée à état chaud, la solution façonnée étant extrudée dans un milieu gazeux sur une zone gazeuse prédéfinie, suite à quoi la solution façonnée extrudée est introduite dans un liquide de coagulation.
tion pour être précipitée, caractérisé en ce que pendant la précipitation et au moins dans la zone de l’entrée de la solution façonnée dans le liquide de coagulation, le liquide de coagulation s’écoule à contre-courant de la direction de déplacement de la solution façonnée.

12. Procédé selon la revendication 11, caractérisé en ce que la précipitation des corps façonnés en cellulose dans un bain de précipitation est réalisée dans un bain de précipitation selon l’une quelconque des revendications 1 à 9.

13. Procédé selon la revendication 12, caractérisé en ce que les corps façonnés en cellulose sont des fibres ou des filaments cellulosiques qui sont encore traités après avoir quitté le bain de précipitation.

14. Procédé selon la revendication 13, caractérisé en ce que les fibres ou filaments cellulosiques qui quittent le bain de précipitation sont d’abord déviés latéralement en contact avec un bord de l’orifice de décharge dudit au moins un ajutage de décharge du bain de précipitation et sont ensuite encore traités.

15. Procédé selon l’une quelconque des revendications 11 à 14, caractérisé en ce que les fils multifilaments sont filés à des débits de 100 à 1 500 m/min et de préférence 800 à 1 200 m/min.