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Process For The Continuous Preparation of Rubber Powders and a Device For Carrying Out The Process

Abstract

Process for the continuous preparation of filler-containing rubber powders, wherein a filler suspension and rubber latex are mixed in the liquid phase. The liquid mixture is passed through a tube [1], at least a coagulant [11] is added to the tube [1] and filler-containing rubber particles are precipitated. The liquid mixture is passed through at least one throttle point [6] of variable throttle cross-section provided in the tube [1]. The throttle cross-section is adjusted with the proviso that the drop in pressure in the liquid mixture achieved with the throttle point [6] is greater than 0.5bar.

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Invention Title:	Process for the Continuous Preparation of Rubber Powders and a Device for Carrying Out the Process

The following statement is a full description of this invention, including the best method of performing it known to me/us:-

Process for the Continuous Preparation of Rubber Powders and A Device for Carrying out the Process

The invention relates to a process for the continuous preparation of filler-containing rubber powders, in which a filler suspension and rubber suspension are mixed in the aqueous phase, the mixture is passed through a tube, at least a coagulant is added in the tube and filler-containing rubber particles are precipitated.

According to the prior art, rubber mixtures are prepared from crude rubber in the form of balls with a high expenditure of time, energy and personnel. The comminution of the balls and mixing with additives are expensive and cost-intensive. On the other hand, the use of free-flowing rubber powders offers the possibility of preparing and processing rubber mixtures simply and quickly.

US 4 265 939 discloses a process for the continuous preparation of rubber powders, but no coagulant is mixed in a tube of relatively narrow cross-section. Effective mixing of the two components is said to take place in the narrow tube on the basis of the turbulent flow. The coagulated rubber material is then passed into a tube of widened cross-section. A polymer, as a coating composition, is fed to the liquid mixture in this tube. This known process leaves something to be desired in respect of effective mixing the components. Furthermore, troublesome deposits or caking on the walls occur, especially in the tube section of narrowed cross-section, and these change the flow circumstances. The properties of the products prepared by this process are therefore not reproducible, and because of the varying flow circumstances during the preparation these products also have disadvantageous undesirable properties. The fact that in the context of a process for the continuous preparation of filler-containing rubber powders the rubber material can be mixed continuously with the filler in a tube is also known in principle from DE 37 23 213 C2 and DE 37 23 214 C2. However, no concrete doctrine on carrying out the process can be obtained from the two documents.

Processes for the preparation of filler-containing rubber powders which are operated batchwise or carried out discontinuously are furthermore known. In these, as a rule a filler-containing suspension is brought together with a suitable latex in the presence of a precipitant and the mixture is then allowed to react in a container. These known processes disadvantageously require a relatively high expenditure on apparatus and time. Such a discontinuous process is described, for example, in DE 28 22 148 02.

In contrast, the invention is based on the object of providing a process in order to prepare a filler-containing rubber powder with reproducible properties without problems and with a low expenditure, and to avoid the disadvantages described above.

According to a first aspect, the present invention consists in process for the continuous preparation of filler-containing rubber powder from aqueous filler dispersions and rubber latices, wherein

a) the filler dispersion and rubber suspension are mixed

b) the liquid mixture is passed through a tube and at least a coagulant is added,

c) the tube comprises at least one throttle point of variable throttle cross-section and optionally one or more inlet points and

d) the throttle cross-section is adjusted, with the proviso that the drop in pressure in the liquid mixture achieved with the throttle point is greater than 0.5bar.

According to a second aspect, the present invention consists in a filler-containing rubber powder prepared by the process of the first aspect.

According to a third aspect, the present invention consists in device for carrying out the process according to the first aspect, wherein a tube for passing a rubber-containing liquid mixture through and for precipitating rubber particles is provided, wherein at least one feed device for rubber material and a feed device for the filler are connected at the start of the tube, wherein furthermore at least one feed device for a coagulant is connected to the tube and wherein at least one throttle organ for variable adjustment of the throttle cross-section is provided in the tube.

According to a fourth aspect, the present invention consists in a device according to the third aspect when used for the continuous preparation of filler-containing rubber powder.

The invention provides a process for the continuous preparation of a filler-containing rubber powder, in which

a) filler dispersions and rubber suspensions are mixed

b) the liquid mixture is passed through a tube, at least one coagulant is added and the filler-containing rubber powder is precipitated,

c) the liquid mixture is passed through a tube with at least one throttle point of variable throttle cross-section,

d) and the throttle cross-section is adjusted, with the proviso that the drop in pressure in the liquid mixture achieved with the throttle point is greater than 0.5bar.

It lies within the context of the invention that the liquid mixture comprises at least one liquid medium, in particular water. Preferably, a suspension, expediently an aqueous suspension of the filler, is employed or added to the rubber material. The rubber material is expediently employed as a rubber latex and/or as a :rubber solution and/or as an aqueous emulsion of a rubber solution. The rubber material may be a synthetic and/or naturally occurring rubber. According to a preferred embodiment of the invention, a styrene-butadiene copolymer is used as the rubber latex. It lies within the context of the invention to employ one or more fillers. Carbon black, for example, is used as the filler. It lies within the context of the invention to add to the rubber material an aqueous suspension of carbon black, in particular a 2 to 7wt% aqueous suspension of carbon black.

The particle size range of the rubber powders obtained according to the invention is in general between 0.05 and 10mm, in particular between 0.5 and 2mm.

The pulverulent rubbers comprise 20 to 250phr, in particular 50 to 100phr of filler (phr: parts per hundred parts of rubber), optionally been modified partly or overall on the surface before the process according to the invention using the organosilicon compounds according to the formulae (I), (II) or (III) known in the rubber sector. The following species, individually or as a mixture with one another, have been found to be suitable rubber types: natural rubber, emulsion SBR with a styrene content of 10 to 50%, butyl-acrylonitrile rubber.

Butyl rubbers, terpolymers of ethylene, propylene (EPM) and non-conjugated dienes (EPDM), butadiene rubbers, SBR, prepared by the solution polymerisation process, with styrene contents of 10 to 25%, and contents of 1,2-vinyl constituents of 20 to 55% and isoprene rubbers, in particular 3,4-polyisoprene.

In addition to the rubbers mentioned, the following elastomers are possible, individually or as a mixture: carboxyl rubbers, epoxide rubbers, trans-polypentenamer, halogenated butyl rubbers, rubbers of 2--chloro-butadiene, ethylene/vinyl acetate copolymers, epichlorohydrins, optionally also chemically modified natural rubber, such as, for example, epoxidised types.

Fillers which are additionally used are in general the carbon blacks and white fillers of a synthetic nature, such as eg. precipitated silicas, or naturally occurring fillers, such as e. g. siliceous chalk, clays etc., known from rubber processing.

Carbon blacks such as are generally employed in rubber processing are particularly suitable.

These include furnace blacks, gas and flame blacks with an iodine adsorption number of 5 to 1000m^2/g, a CTAB number of 15 to 600m^2/g, a DBP adsorption of 30 to 400mL/100g and a DBP number of 50 to 370mL/100g, in an amount of 5 to 250 parts, in particular 20 to 150 parts per 100 parts of rubber, in particular 40 to 100 parts.

The siliceous fillers of synthetic or natural origin known from the rubber sector, in particular precipitated silicas, are likewise suitable.

In general, these have an N_2 surface area, determined by the known BET method, of 35 to 700m^2/g, a CTAB surface area of 30 to 500m^2/g, and a DBP number of 150 to 400mL/100g.

The product according to the invention comprises these silicas in an amount of 5 to 250 parts, in particular 20 to 100 parts, based on 100 parts of rubber.

If the fillers are white naturally occurring fillers , such as clays or siliceous chalks with an N_2 surface area of 2 to 35m^2/g, these are employed in an amount of 5 to 350 parts, based on 100 parts of rubber.

Powders which comprise one or more of the abovementioned fillers in a mixture are also suitable.

In addition to the non-modified fillers of the type mentioned, modified fillers are optionally additionally employed in the preparation of the rubber powders of the invention.

The content of non-modified fillers depends on the mixture specifically to be prepared.

In all cases, the total amount of filler amounts to 20 to 250 phr.

This in general comprises to the extent of 100%, in particular to the extent of 30 to 100%, preferably 60 to 15 100%, the non-modified fillers: silica and/or carbon black.

Organosilicon compounds which are employed for the modification of the surfaces are in general those of the general formulae

$$[(R^1O)_{3-n}Si\text{-}(Alk)_m\text{-}(Ar)_p]_q[B] \text{ (I)}; (R^1O)_{3-n}Si\text{-}(Alk) \text{ (II)}; \text{ or } (R^1O)_{3-n}Si\text{-}(Alkenyl) \text{ (III)}$$

in which the symbols denote

B: -SCN, -SH, -Cl, $-NH_2$ (if q = 1) or $-S_x-$ (if q - 2)

R and R^1 :an alkyl group having 1 to 4 carbon atoms, branched or unbranched, the phenyl radical, where all the radicals R and R^1 in each case can have the same or a different meaning, preferably an alkyl group,

R: a C_1-C_4-alkyl, -C_1-C_4-alkoxy group, branched or unbranched,

n: 0; 1 or 2,

Alk: a divalent straight-chain or branched carbon radical having 1 to 6 carbon atoms,

m: 0 or 1

Ar: an arylene radical having 6 to 12 C atoms

p: 0 or 1, with the proviso that p and m do not simultaneously denote 0,

x: a number from 2 to 8,

alkyl: a monovalent straight-chain or branched unsaturated hydrocarbon radical having 1 to 20 carbon atoms, preferably 2 to 8 carbon atoms,

alkenyl: a monovalent straight-chain or branched unsaturated hydrocarbon radical having 2 to 20 carbon atoms, preferably 2 to 8 carbon atoms.

Modified fillers which are employed according to the invention are described eg. in EP-B 0442 143, EP-B 0177 674, and in particular in the form of granules in EP-A 0795 579 (white fillers), or in EP-B 0519 188 (carbon black)

The bis(alkoxysilylalkyl)-oligosulfanes of the types bis (trialkoxysilylpropyl)-tetrasulfane and -disulfane have proved to be particularly suitable for the pre-modification or the addition to the filler suspension.

The modified fillers known from the applications or patents mentioned and the organosilicon compounds mentioned there are expressly also included in the present application as a constituent of the compositions of the invention.

In addition to the fillers already mentioned, the rubber powders according to the invention in particular comprise known processing or vulcanisation auxiliary substances, such as zinc oxide, zinc stearate, stearic acid, polyalcohols, polyamines, resins, waxes,, plasticiser oils, anti-ageing agents against heat, light or oxygen and ozone, reinforcing resins, flameproofing agents, such as eg. $Al(OH)_3$ and $Mg(OH)_2$, pigments, various crosslinking chemicals and accelerators and optionally sulfur in the concentrations conventional in rubber technology, preferably sulfur in modifications to which surface-active substances have been added such as is commercially available.

The particle size is determined from the filler suspension.

In a particularly preferred form of the process according to the invention, all the solids employed are present in a particle size of <50 µm, preferably <10 µm, before precipitation of the rubber particles out of the suspension. The formation of agglomerates may optionally occur due to the preparation, but these do not adversely influence the processing properties.

The invention also provides a process for the preparation of finely divided, filler-containing rubber powders by precipitation, in a tube provided with throttle flaps at a suitable point, from aqueous mixtures which contain finely divided filler(s) optionally modified with organosilicon compounds, (carbon black and/or siliceous filler) water-soluble salts of a metal of groups IIa, IIb, IIIa and VIII of the periodic table of the elements and a rubber latex or an aqueous emulsion of a rubber solution, optionally in the presence of an organic solvent, which is characterised in that

a) ≥50wt%, but less than 100wt%, of the envisaged amount of the finely divided filler, preferably in the form of an aqueous suspension with a content of 2 to 15wt% in water, optionally with one of the amount envisaged for the modification of the filler surface of one or more of the organosilicon compounds according to the formulae (I), (II) or (III) in an amount of 0.1 to 20wt%, based on the filler, in particular if it is a siliceous filler, preferably precipitated silica, and/or ≥50wt%, but less than 100wt%, of a filler which is at least partly modified on the surface with one or more of the organosilicon compounds (formulae (I), (II) or III), are mixed, in particular in the presence of an emulsifier, with a rubber latex or an aqueous emulsion of a rubber solution and the pH of the mixture is lowered to a value in the range of 7.5 to 6.5, in particular by addition of a Lewis acid, (first stage)

b) the remaining content (splitting content) of the abovementioned finely divided fillers, optionally together with the residual amount envisaged for modification of the filler surface of organosilicon compounds of the formulae (I), (II) or (III), is added in the form of a suspension the pH is lowered, in particular by addition of a Lewis acid, to a value in the range of <6.5 to ~5, preferably ~5.5, so that the rubber in the mixture precipitates completely together with the filler (second stage),

c) the solid which has precipitated is separated off by measures known per se,

d) and is optionally washed and

e) dried.

The organosilicon compounds are employed in particular if siliceous fillers, preferably silicas, are used.

The precipitation process is in general carried out at room temperature, in particular at 20 to 80°C, in the tube, which is preferably provided with several addition points.

The process is carried out by adaptation of the measures known from DE 198 58 706.6.

In the process employed according to the invention, the tube is preferably a tube of circular cross-section. The tube diameter is, for example, 10 to 30mm. The total tube length is preferably 1 to 3m, very preferably 1.5 to 2.5m.

According to the invention, the tube cross-section is reduced at least at one throttle point. Preferably, the diameter of the tube before the throttle point is the same as the diameter of the tube after the throttle point. It lies within the context of the invention that an adjustable throttle organ is located at the throttle point. This organ is preferably a valve, preferably a squeeze valve with an elastic insert. According to a very preferred embodiment of the invention, the throttle organ, of which there is at least one, is set or adjusted with the aid of a drive device. The purpose of the variable reduction according to the invention in the cross-section at the throttle point is so that the continuous process can always be conducted such that the drop in pressure achieved at the throttle point is greater than 0.5bar. In other words, the throttle cross-section is expediently always adjusted with the adjustable throttle organ during the continuous process procedure such that the pressure before the throttle point or before the throttle organ is at least 0.5bar higher than after the throttle point or after the throttle organ. It lies within the context of the invention that the drop in pressure in the liquid mixture achieved with the throttle point is not more than 6bar. Preferably, a drop in pressure of 0.5 to 3bar, very preferably 0.8 to 2.5bar, is achieved at the throttle point. Because of the throttling, a surprisingly optimum mixing of the components passed through the throttle point is achieved. It lies within the context of the invention that the tube cross-section widens again in the direction of flow after a throttle point according to the invention.

According to a very preferred embodiment, the throttle cross-section is varied during the continuous process procedure in order to remove deposits in the throttle region. Deposits in the throttle region means, in particular, deposits or caking on the walls shortly before the throttle point and shortly after the throttle point and in the throttle organ. Thus, if deposits or caking are found in the throttle region, the throttle cross-section is preferably varied as a function thereof. Regulation of the throttle cross-section is expediently automatic.

According to a very preferred embodiment;, a constant drop in pressure is always maintained or set at the throttle point in the context of the process according to the invention. Thus, if the drop in pressure at the throttle point changes because of deposits or caking, the throttle cross-section is preferably readjusted until the desired constant drop in pressure is achieved again. A surprisingly effective mixing can thus be achieved fur the components employed for the preparation of the finely divided rubber powder with the throttling according to the invention, so that end products with the desired properties can be prepared without problems. The fact that; deposits or caking which would disadvantageously change the flow circumstances can be prevented according to the invention in a simple manner and reproducible products can thus be obtained also contributes to this.

According to one embodiment of the invention, the liquid mixture of rubber latex and filler suspension is passed according to the invention through a tube with a throttle point before addition of

a coagulant. In this embodiment, effective premixing of the rubber latex and filler takes place immediately after the throttle point. According to another embodiment of the invention, a liquid mixture of rubber latex, filler and coagulant is passed through one of the abovementioned throttle points. It lies within the context of the invention to provide a first throttle point through which a liquid mixture of rubber material and filler suspension is passed, and thereafter to add coagulant, and only then to pass this liquid mixture through a second throttle point. Effective mixing of the rubber material, filler and coagulant takes place immediately after this second throttle point. According to a preferred embodiment of the invention, both the throttle cross-section of the first throttle point and the throttle cross-section of the second throttle point are adjusted such that the drop in pressure in the liquid mixture achieved with the particular throttle point is greater than 0.5bar and not more than 6bar.

According to a very preferred embodiment., which is of particular importance in the context of the invention, the precipitated rubber particles are coated with a coating composition. It lies within the context of the invention that the coating composition is added to the coagulated rubber material. A filler for rubber is expediently employed as the coating composition. The filler which was already mixed with the rubber material at the start of the process can be employed here. However, it may also be another filler.

According to a preferred embodiment of the invention, the liquid mixture with the added coating composition is passed through an additional throttle point in the tube. The drop in pressure at this throttle point is also expediently more than 0.5bar and not more than 6bar, preferably 3bar.

According to a preferred embodiment of the invention, the liquid mixture is passed through a total of three throttle points in the tube. In this embodiment, the rubber material and filler are first mixed and passed through a first throttle point. In the subsequent section of the tube, the coagulant is added and the resulting liquid mixture is passed through a second throttle point. In the subsequent tube section, the precipitation of rubber particles takes place, and the coating composition is then added in this tube section. The mixture with the coating composition is then passed through a third throttle point. The drop in pressure at each of the three throttle points is preferably more than 0.5bar. At least two, preferably all three, throttle points are expediently constructed as has been explained above for a first throttle point.

According to a second preferred embodiment, two throttle points in total are provided in the tube. In this embodiment, the rubber material, filler and coagulant are first mixed and this liquid mixture is passed through the first throttle point. In the tube section after the first throttle point, the precipitation of the rubber particles then takes place, and the coating composition is subsequently added. The mixture with the coating composition is then passed through a second throttle point. The throttle cross-section at each of the two throttle points is preferably adjusted with the proviso that the drop in pressure in the liquid mixture achieved with the particular throttle point is greater than 0.5bar and not more than 6bar. The two throttle points are expediently constructed as has been explained above for a first throttle point.

The invention also provides a device wherein a tube for passing a rubber-containing liquid mixture through and for precipitating rubber particles is provided, wherein at least one feed device for rubber material and a feed device for the filler are connected at the start of the tube, wherein

furthermore at least one feed device for a coagulant is connected to the tube and wherein at least one throttle organ for variable adjustment of the throttle cross-section is provided in the tube.

It is preferred that at least once feed device for a coating composition is connected to the tube in the direction of flow after the throttle organ.

It is further preferred that an additional throttle organ for variable adjustment of the throttle cross-section is provided in the direction of flow after the feed device for the coating composition.

The invention is based on the finding that a filler-containing rubber powder with reproducible properties can be obtained in a manner which is not particularly expensive if the continuous procedure according to the invention is used. A surprisingly effective mixing of the components employed here (rubber material, filler, optionally coagulant) is achieved with the throttle points according to the invention, so that thereafter the desired reaction proceeds completely and without problems. In the case of the components in question here in particular, trouble-free mixing as a rule presents problems, for example in order to achieve a completely homogeneous embedding of the filler in the rubber. With the variable throttling according to the invention, this mixing is achieved in a manner which, to the expert, is surprisingly effective and functionally reliable. A specific turbulent flow is generated with the aid of the throttle organs, preferably throttle valves, because of the drop in pressure according to the invention, and as a result a very homogeneous mixture is achieved, so that homogeneous end products also result. This was not possible with the known processes described above. In addition to optimum mixing of the components, as a result of the throttling according to the invention deposits or caking in the region of the throttle point can furthermore be avoided surprisingly effectively by changing the throttle cross-section. By regulating the pressure loss with the aid of variable throttle cross--sections, throttle points which are insensitive to blocking and function as optimum mixing organs are thus obtained according to the invention. Pulverulent rubbers which can already comprise all the rubber auxiliary substances can be prepared by the process according to the invention. In addition to fillers, these auxiliary substances are, for example, sulfur, vulcanisation accelerators, activators and rubber anti-ageing agents. These rubber auxiliary substances can be added to the liquid mixture without problems during the continuous process procedure, and these rubber auxiliary substances are then present in the pulverulent end products in a very homogeneous distribution. In contrast to the known processes, after-treatments, in particular coating of the rubber particles, are also possible in a simple manner in that a further throttle organ is provided according to the invention and the drop in pressure established at this throttle organ contributes towards effective mixing also of the coating composition with the liquid mixture and thus functionally reliable and uniform coating of the rubber particles. With the continuous procedure in the tube according to the invention, considerably higher yields per unit time are achieved compared with a discontinuous procedure. The residence times of the liquid mixture in the tube according to the invention are relatively short.

The invention is explained in more detail below with the aid of a drawing which merely shows one embodiment example. The diagrams show:

Fig. 1 a first embodiment of a device for carrying out the process according to the invention,

Fig. 2 a diagram of the device according to figure 1, in which the pressure is shown schematically as a function of the tube length or of the residence time in the tube and

Fig. 3 a second embodiment of the subject matter according to figure 1.

Figures 1 and 3 show a device for the continuous preparation of a filler-containing rubber powder. The device comprises a tube 1 for passing a rubber-containing liquid mixture through and for precipitation of rubber particles. Both in the embodiment example according to figure 1 and in the embodiment example according to figure 3, a feed device 2 for rubber material 3 and a feed device 4 for filler 5 are connected to the start of the tube 1. The rubber material may be a rubber latex, preferably a styrene/butadiene latex. The filler is an aqueous suspension of carbon black.

In the embodiment according to figure 1, the liquid mixture of rubber material 3 and filler 5 is first passed through the first throttle organ 6. The throttle cross-section of this first throttle organ 6 is always adjusted such that the drop in pressure in the liquid mixture achieved at the throttle point is greater than 0.5bar. It goes without saying that corresponding pressure sensors not shown in the figures are provided. In the embodiment according to figure 1, the particular pressure conditions which exist in the individual tube sections can be read of via pressure displays 7, 8, 9. The process is preferably carried out such that a constant drop in pressure always remains established at the first throttle organ 6 during the continuous process procedure. The throttle cross-section is expediently always readjusted such that this constant drop in pressure exists. According to a preferred embodiment, the readjustment of the first throttle organ 6 rakes place automatically. In the embodiment example, the first throttle organ 6 may be constructed as a squeeze valve. Preferably and in the embodiment example according to figure 1, the tube section before the first throttle organ 6 has the same cross-section as the tube section after the first throttle organ 6. A particularly effective mixing of the rubber material with the filler takes place due to the throttling at the first throttle organ 6.

A coagulant 11 is added to the liquid mixture in the tube 1 via a feed device 10. This liquid mixture is then passed through a second throttle organ 12, the throttle cross-section of which is adjusted such that the drop in pressure in the liquid mixture achieved with the throttle point is greater than 0.5bar. With this second throttle organ 12 also, the throttle cross-section is expediently always adjusted during the process such that a constant drop in pressure exists. Preferably, this second. throttle organ 12 is also readjusted automatically as a function of the drop in pressure measured. Preferably and in the embodiment example, the tube diameter before the second throttle organ 12 corresponds to the tube diameter after the second throttle organ 12. After the second throttle organ 12, the coagulant 11 is mixed surprisingly effectively with the liquid mixture. In the tube section 13 after the second throttle organ 12, the intensively mixed reactants then react and the rubber particles precipitate here.

Preferably and in the embodiment example according to figure 1, a further feed device 14 for a coating composition 15 is provided. In the embodiment example, the coating composition 15 may correspond to the filler 5. The previously precipitated rubber particles are to be coated with this coating composition 15. In the embodiment example according to figure 1, the liquid mixture with the coating composition 15 is passed through an additional throttle organ 16. In this additional throttle organ 16 also, the throttle cross-section is adjusted with the proviso that the drop in pressure in the liquid mixture achieved with the throttle point is greater than 0.5bar. Preferably, a constant drop in pressure is also always established here, and as in the case of the throttle organs 6, 12 described

above, this drop in pressure is preferably readjusted automatically. The tube diameter before the additional throttle organ 16 expediently corresponds to the tube diameter after the additional throttle organ 16. After the additional throttle organ 16, the coating composition is mixed surprisingly effectively with the liquid mixture. In the tube section after the additional throttle organ 16, coating of the rubber particles then takes place. The precipitation product 17 is taken off at the end of the tube in the form of coated rubber particles. Separation of the coated filler-containing rubber particles from the liquid phase or from the aqueous phase then expediently takes place, and drying of the rubber particles is then preferably carried out. A finely divided rubber powder with reproducible properties is obtained.

In figure 2, for a device according to figure 1, the pressure P in the tube 1 is plotted against the tube length (in metres) and against the residence time of the liquid mixture in the tube (in seconds). The tube length here is measured from the first throttle organ 6. According to this, a tube with a length of 2.5m (measured from the first throttle organ 6) is employed in the embodiment example. The tube diameter is 25mm in the embodiment example. The diagram according to figure 2 shows that the drop in pressure ΔP_1 at the first throttle organ 6 is 1.5bar. The drop in pressure ΔP_2 at the second throttle organ 12 is 2bar in the embodiment example, and the drop in pressure ΔP_3 at the additional throttle organ 16 is 1bar. Overall, the pressure of the liquid mixture in the tube 1 has thus fallen from an initial pressure of 4.5bar to 0bar in this embodiment example. The maximum initial pressure is preferably 6bar. According to a preferred embodiment, the drop in pressure ΔP_1 at the first throttle organ 6 is 0.5 to 2.5bar, preferably 1 to 2bar. Preferably, the drop in pressure ΔP_2 at the second throttle organ 12 is 0.5 to 3bar, preferably 1.5 to 2.5bar. The drop in pressure ΔP_3 at the additional throttle organ 16 is expediently 0.5 to 2bar, preferably 0.5 to 1.5bar.

Figure 3 shows a further embodiment of a device according to the invention. Rubber material 3 in the form of a 20wt% latex of styrene/butadiene copolymer is fed in via the feed device 2. Filler 5 in the form of a 3wt% aqueous carbon black suspension is fed to the tube 1 via the feed device 4. For premixing of the rubber material 3 with the filler 5, the liquid mixture is first passed through a baffle plate arrangement 18, and thereafter coagulant 11 in the form of a 20wt% aluminium sulfate solution is fed to the tube 1 via the feed device 10. The liquid mixture with the coagulant is then passed through a first throttle organ 6. The throttle cross-section of this first throttle organ 6 is adjusted such that the drop in pressure achieved in the liquid mixture is greater than 0.5bar. A very effective mixing of the rubber material, filler and coagulant takes place after the first throttle organ 6. Acid 20, preferably a 10wt% sulfuric acid solution, is fed to the tube 1 via the feed device 19. A pH of between 1 and 10 is established here. It lies within the context of the invention to establish a pH of between 2.5 and 9. A pH of greater than 6 is preferably established. The pH can be checked with the pH display 21. The size of the coagulated or filler-containing rubber particles can be controlled by the pH adjustment. In the tube section in the direction of flow after the feed device 19, precipitation of the rubber particles then takes place. Coating composition 15 is fed in via the feed device 14. The liquid mixture with the coating composition 15 is then passed through the additional throttle organ 16. A very effective mixing and therefore an effective coating of the rubber particles takes place after the additional throttle organ 16. Thereafter, the finished precipitation product 17 is taken off in the form of

coated finely divided rubber particles present in the aqueous phase. The rubber particles are then separated from the aqueous phase and the rubber powder is subsequently preferably dried.

In the embodiment example according to figure 3, the total tube length is 1.5m. The tube section between the feed devices 2 and 4 and the first throttle organ 6 has a length of 0.25m. The length of the tube section between the first throttle organ 6 and the additional throttle organ 16 is 1.0m, and the tube section after the additional throttle organ 16 has a length of 0.25m. The diameter of the tube 1 outside the throttle organs 6, 16 is 15mm.

The claims defining the invention are as follows:

1. Process for the continuous preparation of filler-containing rubber powder from aqueous filler dispersions and rubber latices, wherein

a) the filler dispersion and rubber suspension are mixed

b) the liquid mixture is passed through a tube and at least a coagulant is added,

c) the tube comprises at least one throttle point of variable throttle cross-section and optionally one or more inlet points and

d) the throttle cross-section is adjusted, with the proviso that the drop in pressure in the liquid mixture achieved with the throttle point is greater than 0.5bar.

2. Process according to claim 1, wherein the throttle cross-section is varied during the continuous process procedure to remove deposits in the throttle region.

3. Process according to claim 1 or claim 2, wherein the liquid mixture of rubber latex and filler is passed through the throttle point before addition of a coagulant.

4. Process according to claim 1 or claim 2, wherein a liquid mixture of rubber latex, filler and coagulant is passed through the throttle point.

5. Process according to any one of claims 1 to 4, wherein the precipitated rubber particles are coated with a coating composition.

6. Process according to claim 5, wherein the liquid mixture with the coating composition is passed through an additional throttle point in the tube.

7. Process for the continuous preparation of filler-containing rubber powder from aqueous filler dispersions and rubber latices, said process being substantially as hereinbefore described with reference to the accompanying drawings.

8. A filler-containing rubber powder prepared by the process of any one of claims 1 to 7.

9. Device for carrying out the process according to any one of claims 1 to 7, wherein a tube for passing a rubber-containing liquid mixture through and for precipitating rubber particles is provided, wherein at least one feed device for rubber material and a feed device for the filler are connected at the start of the tube, wherein furthermore at least one feed device for a coagulant is connected to the tube and wherein at least one throttle organ for variable adjustment of the throttle cross-section is provided in the tube.

10. Device according to claim 9, wherein at least one feed device for a coating composition is connected to the tube in the direction of flow after the throttle organ.

11. Device according to claim 10, wherein an additional throttle organ for variable adjustment of the throttle cross-section is provided in the direction of flow after the feed device for the coating composition.

12. Device according to any one of claims 9 to 11, wherein at least one feed device for an acid medium for pH adjustment in the liquid medium is connected to the tube.

13. Device for the continuous preparation of filler-containing rubber powder from aqueous filler dispersions and rubber latices, said device being substantially as hereinbefore described with reference to the accompanying drawings.

14. A device according to any one of claims 9 to 13 when used for the continuous preparation of filler-containing rubber powder.

Dated 2 May, 2003
PKU Pulver Kautschuk Union GmbH

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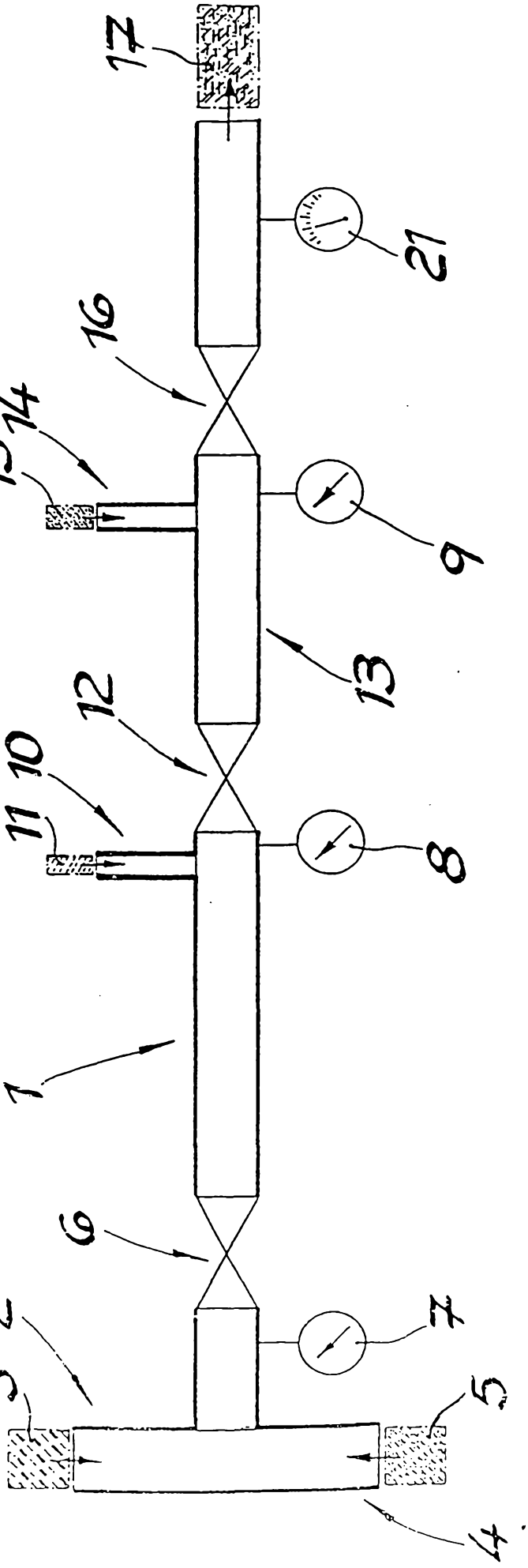
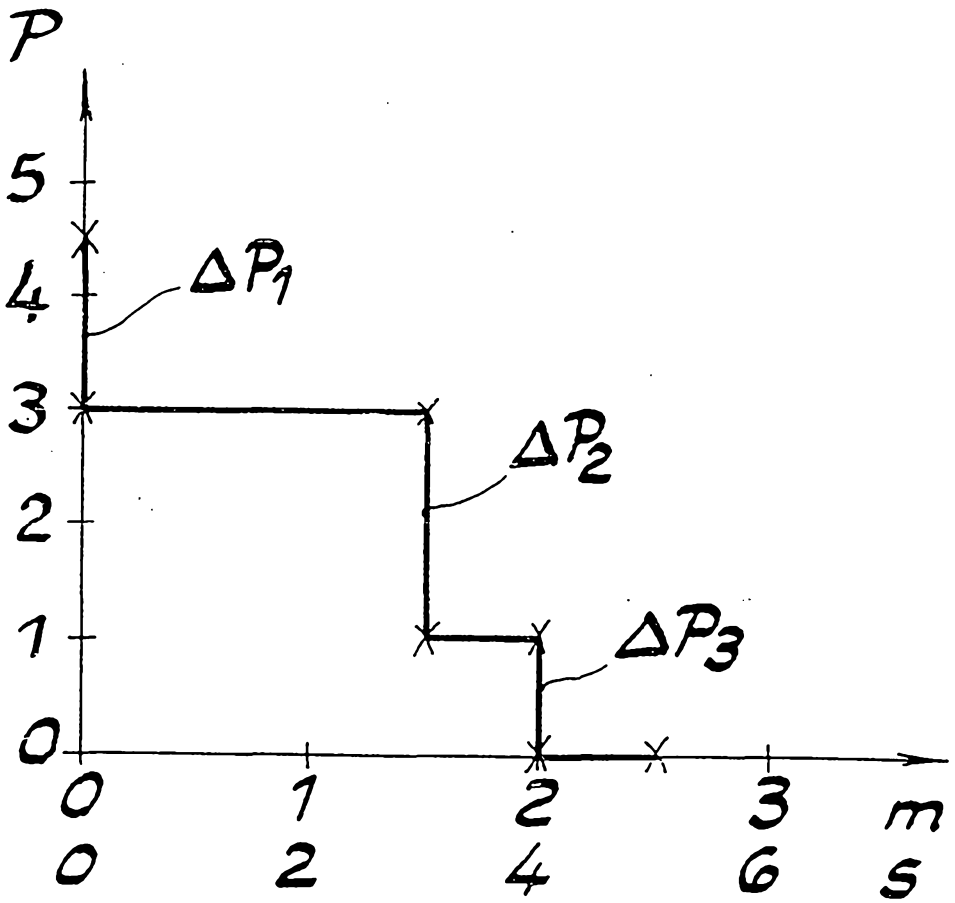


Fig.1

Fig.2



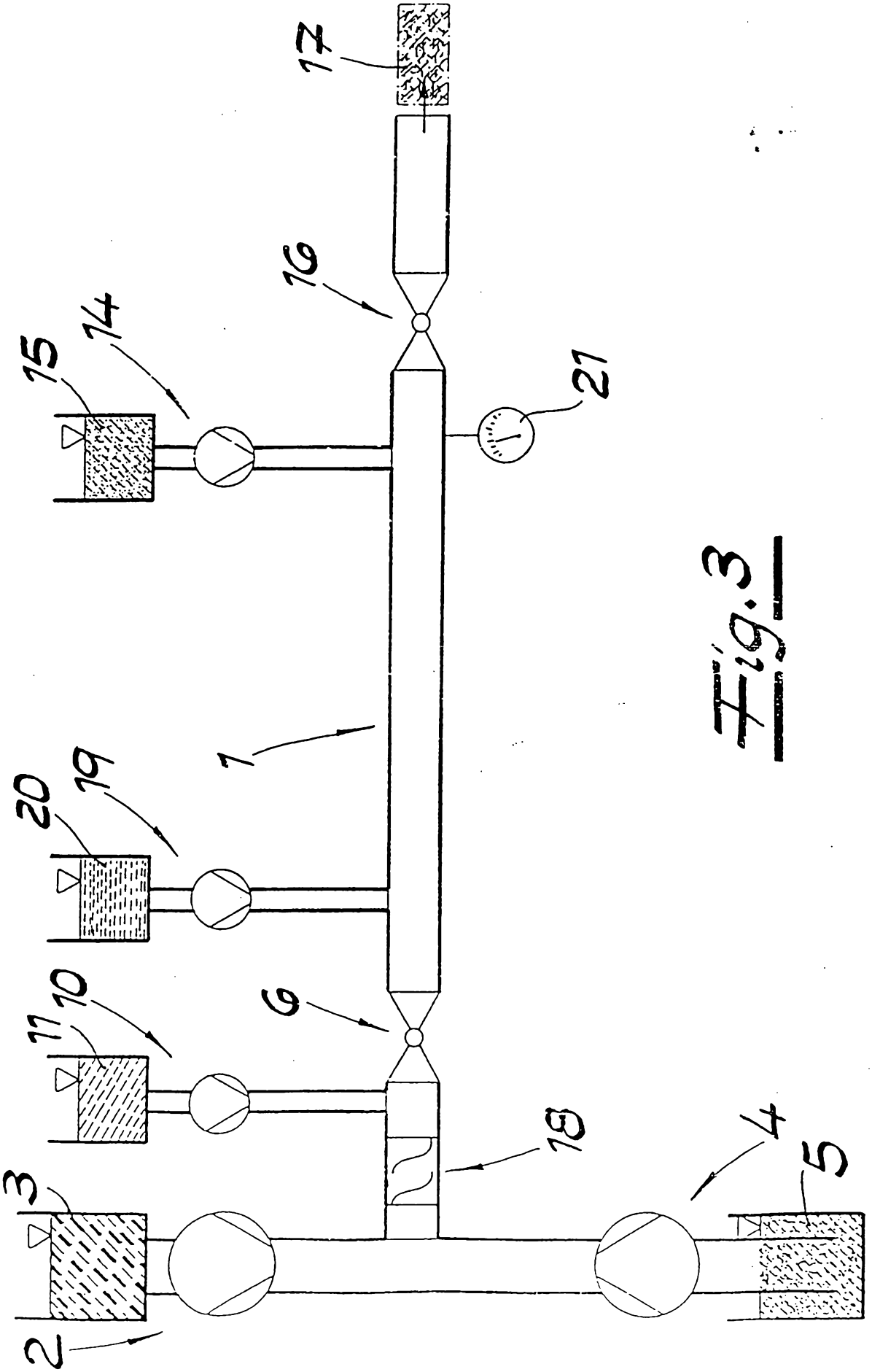


Fig. 3