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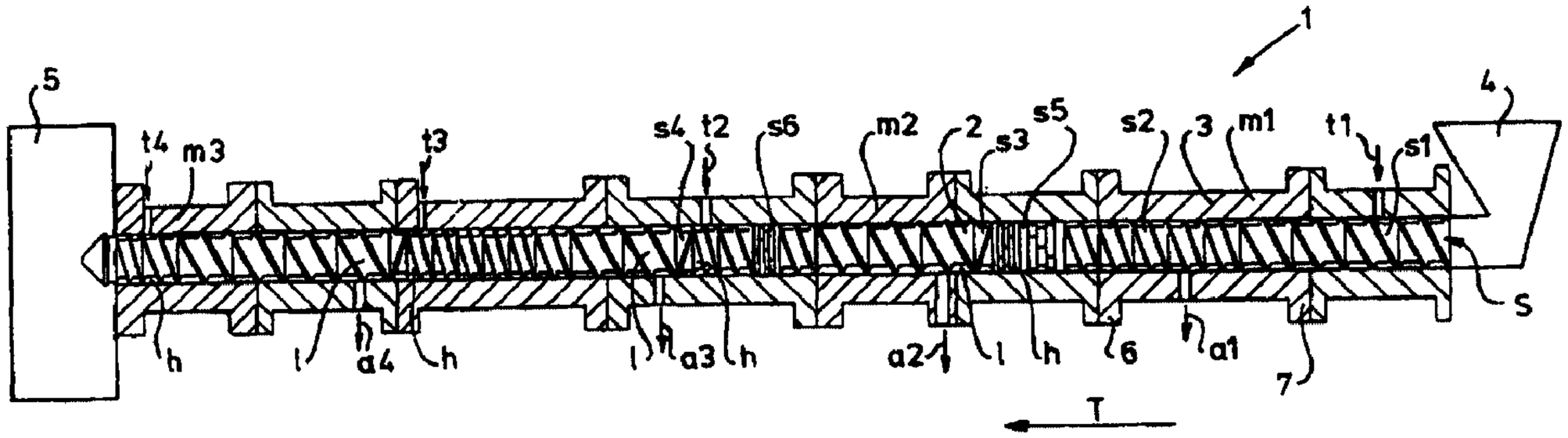
(54) Titre : PROCEDE D'EXTRACTION D'UNE SUBSTANCE A PARTIR D'UNE MATIERE DE DEPART ET APPAREIL POUR REALISER LE PROCEDE

(54) Title: METHOD FOR THE EXTRACTION OF A SUBSTANCE FROM A STARTING MATERIAL AND EXTRACTION APPARATUS FOR CARRYING OUT THE METHOD



(57) Abrégé/Abstract:
The invention relates to a counter-current extraction process with which the starting material to be extracted is conveyed in an extruder through zones of high and low pressure. The zones of high and low pressure can, for example, be formed by different screw elements of opposing pitch. Extractant is fed in in the high pressure zones which are located upstream of the screw elements of opposing pitch. The extractant then flows in counter-current to a discharge opening which is located in or close to the low pressure zone, downstream of a screw element of opposing pitch. With the extraction method according to the present invention a stable counter-current extraction process can be obtained over a very short extraction length and a high extraction yield can be achieved within a short time by intensive mixing of the starting material with the extractant. Furthermore, high pressures can be used in the extraction apparatus according to the present invention since the discharge openings are located close to the low pressure zones. As a result the extraction yield is further increased and extraction fluids in supercritical state can be used.

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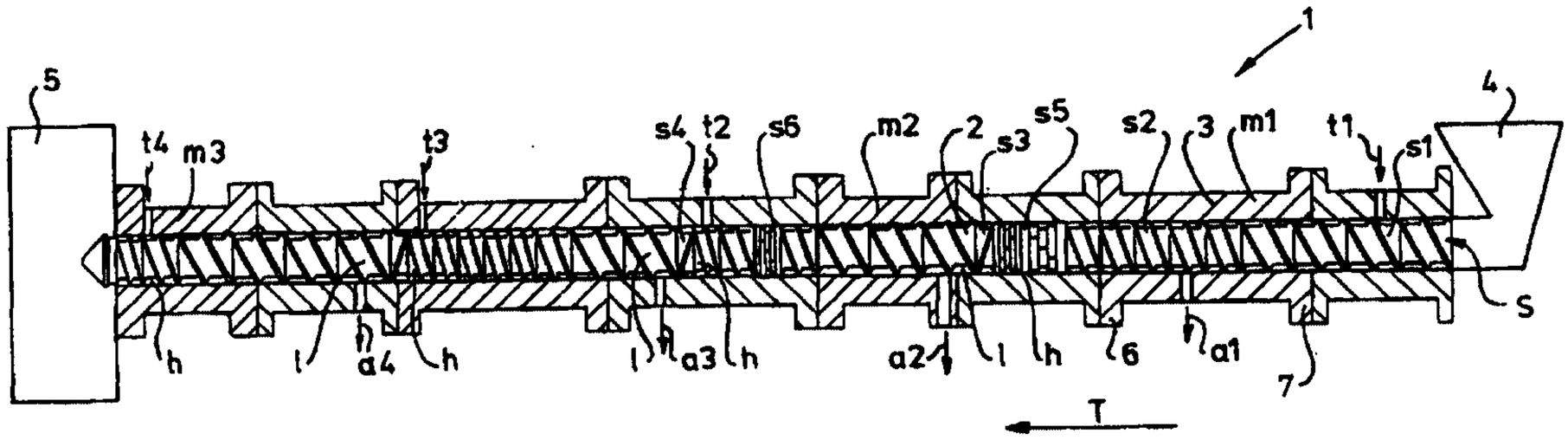
(54) Title: METHOD FOR THE EXTRACTION OF A SUBSTANCE FROM A STARTING MATERIAL AND EXTRACTION APPARATUS FOR CARRYING OUT THE METHOD



(57) Abstract

The invention relates to a counter–current extraction process with which the starting material to be extracted is conveyed in an extruder through zones of high and low pressure. The zones of high and low pressure can, for example, be formed by different screw elements of opposing pitch. Extractant is fed in in the high pressure zones which are located upstream of the screw elements of opposing pitch. The extractant then flows in counter–current to a discharge opening which is located in or close to the low pressure zone, downstream of a screw element of opposing pitch. With the extraction method according to the present invention a stable counter–current extraction process can be obtained over a very short extraction length and a high extraction yield can be achieved within a short time by intensive mixing of the starting material with the extractant. Furthermore, high pressures can be used in the extraction apparatus according to the present invention since the discharge openings are located close to the low pressure zones. As a result the extraction yield is further increased and extraction fluids in supercritical state can be used.

Method for the extraction of a substance from a starting material and extraction apparatus for carrying out the method

The invention relates to a method for the extraction of a substance from a starting material, wherein the starting material is brought together with an extraction fluid in an extruder, wherein the starting material is moved along an extrusion channel in a conveyor device and wherein the extruder contains at least two pressure-raising elements which are located apart, with a low pressure zone between said elements and a high pressure zone located downstream thereof.

In the foodstuffs industry organic waste streams and specific product streams frequently contain high value components. These components are usually extracted from the product streams batchwise. With the known techniques the starting material is usually pressed, followed by precipitation, washing out by means of boiling or by means of a belt press, large quantities of extraction fluid being used.

It is also known to produce bleached chemical-mechanical wood pulp from wood chips by feeding the latter through a twin screw extruder such as that manufactured by Clextral under the trade name Bivis. In this process a number of screw elements with opposing pitch (reverse screw elements) are used in the casing of the extruder in order to form high pressure zones for intensive kneading of the product. Chemicals such as NaOH and $NaHSO_3$ as well as steam are injected into the casing of the extruder and discharged from the extruder in co-current with the starting material.

A process of this type has the disadvantage that it is not optimum for extraction and that the starting material can be forced out of the casing of the extruder under pressure at the tapping points. As a result the pressures which can be used are limited and the concentrations of substances which can be extracted in an extraction process are usually relatively low.

FR 2 619 514 describes an extraction process using a twin screw extruder, wherein extraction takes place at relatively low pressures in a number of co-current processes connected in series.

WO 90/10484 discloses an extraction method with which the starting material is moved by means of a single screw extruder in counter-current with a solvent. With the known apparatus having a single screw, expression of the starting material with extractant in the compression zone is possible to only a limited extent since if too much fluid is used no further solid is transported. Extraction under high pressure is also not possible in the

known apparatus, whilst the flow of the starting material and the extractant are highly integrated, so that the extraction proceeds relatively inefficiently.

It is therefore an aim of the present invention to provide an extraction process with which relatively high pressures can be used, with which very good mixing of starting material and extraction fluid takes place, with which a high yield is achieved and with which the risk of starting material being forced out of the casing through the discharge openings is reduced.

A further aim of the present invention is to provide an extraction process which can be operated continuously and with which various substances can be extracted from a starting material with high selectivity.

To this end the method according to the present invention is characterised in that the extraction fluid is fed to the extruder via a feed opening in or close to the high pressure zone, is then brought to the low pressure zone in counter-current to the direction of transport of the starting material and is discharged from the extruder via a discharge opening in or close to the low pressure zone, with the starting material being continually supplied to the low pressure zone in the direction of transport and with continuous discharge of the starting material from the high pressure zone in the direction of transport.

According to the invention, the extraction fluid is supplied to the extruder and fed in counter-current over a continuous supply of the starting material through the extrusion channel. Surprisingly, it has been found that the material to be extracted is highly comminuted in the high pressure zones, so that a very high surface area/volume ratio is obtained and very good mixing with the extraction fluid (solvent) takes place, as a result of which efficient extraction is possible by means of counter-current, with a very high extraction yield. By placing the discharge openings for the extraction fluid in or close to the low pressure zones it is also possible to raise the pressure in the extraction apparatus to relatively high levels, such as, for example, higher than 10 bar, without the starting material being forced out through the discharge openings. At these high pressures the solubility in the extraction fluid is high and extraction can take place efficiently. At the high pressures the substance to be extracted can be dissolved more rapidly by passing through an arbitrary solvent in counter-current, such as, for example, water or a salt solution. By means of separate discharge of the extraction fluid from the extruder, while the starting material is transported further along the extrusion channel, a high grade substance, such as an oligomer or polymer condensate, can be obtained efficiently and

continuously from a low grade residual stream or waste stream. The extracted, insoluble comminuted residual fraction can, for example, be further transported to a second low pressure and high pressure zone, an extraction fluid once again being supplied, which extraction fluid is discharged close to the second low pressure zone. By this means a further substance can be extracted from the starting material, with a relatively high selectivity. The properties of the extracted product can be controlled by the selection of the extraction fluid used, the specific pressures and temperatures in the extruder, the starting material/extraction fluid ratio, contact time and the like.

Examples of extracted substances include secondary metabolites, such as odour substances, colorants and flavourings, for example carvone and limonene from caraway seeds, vanilla from vanilla beans, or pentosans from wheat waste, pectin from the skins of citrus fruits and inulin from chicory. Furthermore, the method according to the invention can also be used for non-organic materials, such as extraction of catalyst residues from polymers. With the method according to the present invention it is also possible advantageously to use an extraction fluid in the supercritical state, such as CO_2 at 75 bar and 35 °C, as extraction fluid. The supercritical state can be maintained over the entire extraction length.

In one embodiment of the method according to the invention, after the extrusion step the extruded starting material is fed to drying or press means. By this means the starting material can be processed to give, for example, fibreboard, cattle feed pellets and covering for a stall floor.

The extraction apparatus according to the invention comprises an extruder, for example a multi-screw extruder, with, in or close to the low pressure zone, a discharge opening in the wall of the extrusion channel for discharging the extraction fluid. The discharge opening is provided with screening means. A feed opening is made in the wall of the extrusion channel in or close to the high pressure zone located downstream of the low pressure zone.

Preferably, the pitch of the screw in the extruder is relatively large in the low pressure zones and is relatively small in the high pressure zones. In this way the pressure in the extruder can be controlled in a simple manner by adjusting the pitch of the screw. It is also possible to create high pressure zones and low pressure zones by locally reversing the pitch of the screw with respect to the pitch of the transport part of the screw.

The extraction apparatus according to the invention is of modular construction and

is made up of modules which can be coupled to one another. The modules can be coupled to one another by means of coupling elements such as external flanges which can be connected to one another. By adjusting the number of modules, various substances can be extracted from the starting material as required with the aid of a number of different extraction fluids. The strength of the dynamic seals which are formed by the screw elements with opposing pitch and the pressure which these seals are able to withstand depend on the geometry of the screw element and on the rheological properties (the way in which the material deforms under the influence of a force) of the material with which the extruder is filled. The rheological properties can be adjusted by mixing the starting material with one or other plasticiser or lubricant, such as, for example, in the case of caraway seeds as the starting material where the addition of a small amount of water has a substantial influence on the properties in the seals.

The invention will be explained in more detail with reference to the single appended figure.

The figure shows an extraction apparatus 1 having an extrusion channel 2 surrounded by a cylindrical wall 3. Two screws S, which partially engage with one another and have screw elements or modules S_1, S_2 of different pitch, have been placed in the extrusion channel 2. Screw elements S_3, S_4 having a pitch which is opposite to the pitch of the screw elements S_1, S_2 have also been accommodated in the extrusion channel 2. Kneading elements S_5, S_6 can be accommodated between the screw elements. By means of the screw elements S_1, S_2 of different pitch, the kneading elements S_5, S_6 and the screw elements S_3, S_4 of opposing pitch, high pressure zones h are formed in the extrusion channel 2. The high pressure zones h are each located upstream of the screw elements S_3, S_4 of opposing pitch, in the vicinity thereof. Zones of a relatively low pressure are located in the extrusion channel 2 in positions which are indicated by reference symbol "l", in each case downstream of the screw elements S_3, S_4 of opposing pitch.

Feed openings t_2, t_3 and t_4 have been made in the wall 3 at the location of the high pressure zones h. The extraction fluid can be introduced by means of an injection device through the feed openings t_2, t_3 and t_4 into the extrusion channel 3. Discharge openings a_2, a_3 and a_4 have been made in the wall 3 at the location of the low pressure zones l. The extraction fluid introduced through a feed opening t_2, t_3 and t_4 moves in counter-current over the material moved by the screw S in the direction of transport T to a respective discharge a_2, a_3 and a_4. With this arrangement an effective extrusion space is formed

between each two screw elements S_3, S_4 of opposing pitch located some distance apart. Because the discharge openings a_2, a_3 and a_4 are located close to the low pressure zone of such an extrusion space, the pressures in the extrusion space can be raised relatively high without the transported material being forced out through the discharge openings. As a result of the high pressures, efficient extraction is possible because the solubility in the extractant is relatively high at these pressures. The discharge openings a_2, a_3 and a_4 are each provided with a screening device, so that only the extraction fluid is able to pass through them, whilst the starting material remains in the extrusion channel 2.

The starting material can be fed to the extrusion channel 2 via a filling chute 4. A reactant or extraction fluid can already be added to the starting material at the location of the filling chute 4. It is also possible to carry out a pretreatment of the starting material in a multi-current extraction step by adding a reactant through feed opening t_1, and discharge of said reactant through discharge opening a_1. A drying installation or press installation 5, in which fibreboard, pellets and the like can be formed from the extracted residual material, is located at the end of the extraction apparatus 1.

The length of the extraction apparatus as shown in Figure 1 is, for example, 2 metres for a diameter of 44 mm. Preferably, the apparatus of modular construction with modules M_1, M_2, M_3 which can be coupled to one another. Each module M_1, M_2, M_3 has external flanges 6, 7 which, by means of bolts which are not shown in the figure, can be coupled to similar flanges of an adjacent module. Each module M_1, M_2 has a conveyor screw and/or a screw element of opposing pitch and/or a kneading element and can be provided with a feed opening or a discharge opening in the wall 3. By use of the extraction apparatus described above an open system is obtained in which very high pressures can nevertheless be achieved.

In order to illustrate the effectiveness of the extraction process according to the present invention, an extraction of sugars from chicory was carried out with the aid of a Clextral model BC45 co-rotating twin screw extruder.

The screw construction is modular and in accordance with the principles as shown in the single figure. An extruder was constructed from 14 separate screw elements according to Table 1:

TABLE 1

Element no.	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Screw type	TZ	TZ	TZ	TZ	RSE	RSE	TZ	TZ	TZ	TZ	RSE	RSE	RSE	Ring
Length [mm]	100	50	100	100	50	50	200	200	100	50	50	50	50	100
Pitch [mm]	50	35	25	15	-25	-15	50	35	35	35	-25	-25	-15	-
Slot size [mm]					10	8					4	6	12	

The screw type used is indicated by TZ, which indicates a screw element of trapezoidally shaped pitch. The term RSE relates to a reverse screw element (or a screw element having an opposingly directed pitch, such as the elements S_3, S_4 in the figure). The term Ring relates to a neutral element without a conveyor action. Each reverse screw element can be provided with a hole, the diameter of which is shown in the fifth row of Table 1. The inlet opening for injection of the extraction fluid, or solvent, was at the level of the tenth screw element, having a pitch of 35 mm. The upstream discharge opening was at the seventh screw element, having a pitch of 50 mm. The length of the extraction path, or the distance between the feed opening and the discharge opening, is approximately 50 cm. A discharge opening for a first expression of the raw material without the addition of extraction fluid or solvent is located further upstream, at the level of the first screw element, having a pitch of 50 mm.

With the aid of such a configuration it is possible to achieve a stable counter-current extraction process at a speed of 25 revolutions per minute, with an amperage of 8 ampère and a pressure at the extruder outlet of 2 bar. The temperature of the extruder wall at the feed and the discharge was 25 °C and 32 °C respectively. With these settings 210.00 g/min chicory (i.e. 186.90 g/min water and 23.10 g/min solids) were fed into the extruder. 125.66 g/min chicory (i.e. 116.49 g/min water and 9.17 g/min solids) were removed in the first expression of the raw material. The remaining 84.34 g/min chicory was brought into contact with 125.00 g/min water in counter-current in the extraction zone. The component balance is given in Table 2. The sugars were analysed for molecular size (monosaccharides, disaccharides, trisaccharides, etc.) with the aid of HPLC.

TABLE 2

		First expression		Extraction zone				Total process	
		Chicory in [g/min]	Chicory out [g/min]	Chicory in [g/min]	Water in [g/min]	Out through discharge opening [g/min]	Residue out [g/min]	Total in [g/min]	Total out [g/min]
Water		186.90	116.49	70.41	125.00	103.14	86.73	311.90	306.36
Solids		23.10	9.17	13.93	-	2.86	11.27	23.10	23.31
Sugars	1	5.00	3.25	1.74	-	1.05	0.52	5.00	4.82
	2	2.69	1.57	1.12	-	0.53	0.30	2.69	2.40
	3,4	1.58	1.19	0.38	-	0.34	0.09	1.58	1.62
	5,6	0.90	0.63	0.27	-	0.23	0.13	0.90	0.99
	7,8,9	0.18	0.15	0.03	-	0.08	0.03	0.18	0.27
Total sugars		10.33	6.80	3.53	-	2.24	1.07	10.33	10.10

The yield from the first pressing step (the ratio of the quantity of expressed sugars to the quantity of sugars in the raw material) is 65%. The yield in the extraction zone (the ratio of extracted sugars to the quantity fed into the extraction zone) is 63%. The total yield of the process (the total quantity of sugars obtained to the quantity of sugars in the raw material) is 88%.

In view of the short length over which the counter-current extraction takes place, the extruder described is a highly efficient extractor.

Claims:

1. Method for the extraction of a substance from a starting material, wherein the starting material is brought together with an extraction fluid in an extruder, wherein the starting material is moved along an extrusion channel in a conveyor device and wherein the extruder contains at least two pressure-raising elements which are located apart, with a low pressure zone and a high pressure zone located downstream of said low pressure zone and between said elements, characterized in that the extraction fluid is fed to the extruder via a feed opening in or close to the high pressure zone, is brought to the low pressure zone in counter-current to the direction of transport of the starting material and is discharged from the extruder via a discharge opening in or close to the low pressure zone, with the starting material being continually supplied to the low pressure zone in the direction of transport and with continuous discharge of the starting material from the high pressure zone in the direction of transport.

2. Method according to Claim 1 characterized in that at least a second low and a high pressure zone are located downstream with respect to the first low and high pressure zones, wherein an extraction fluid is fed to the extruder in or close to the second high pressure zone and wherein the extraction fluid is discharged from the extruder in or at the second low pressure zone.

3. Method according to Claim 1 or 2, characterized in that, following extraction, the starting material is fed from the extruder to drying and/or press means.

4. Method according to any one of Claims 1 to 3, characterized in that the starting material is an organic material.

5. Extraction apparatus (1) for separating a substance from a starting material, comprising an extruder having a feed (4), an extrusion channel (2) a screw (S) located in the extrusion channel and at least two pressure-raising elements which are located apart, with a low pressure zone (l) and a high pressure zone (h) located downstream of the low pressure zone and between said elements, characterized in that in or close to the high pressure zone (h) a feed opening (t_2, t_3, t_4) is made in the wall (3) of the extrusion channel (2) for supplying the extraction fluid and in that in or close to the

low pressure zone (l) a discharge opening (a_2, a_3, a_4) is made in the wall (3) of the extrusion channel (2) for discharging the extraction fluid supplied, the discharge opening (a_2, a_3, a_4) being provided with screening means through which the starting material in the extrusion channel is not able to pass.

6. Extraction apparatus according to Claim 5, wherein the pitch of the screw (S) is greater in the low pressure zone (l) than in the high pressure zone (h).

7. Extraction apparatus according to Claim 5 or 6, wherein the pitches of screw components on either side of a high pressure zone (h) are mutually opposed.

8. Extraction apparatus according to any one of Claim 5 to 7, comprising at least a further high and low pressure zone, downstream of the first high and low pressure zones, and at least one further feed opening for an extraction fluid in or close to the further high pressure zone and a further discharge opening in or close to the further low pressure zone.

9. Extraction apparatus according to Claim 5, 6, 7 or 8, characterized in that the apparatus is constructed of modules (M_1, M_2) which can be connected to one another, each having a high pressure zone (h) and a low pressure zone (l) and at least on of a feed opening and a discharge opening, as well as coupling elements (6, 7) for connecting the modules to one another.

10. Extraction apparatus according to any one of Claims 5 to 9, characterized in that each feed opening (t_2, t_3, t_4) is provided with an injection device for injecting the extraction fluid under pressure.

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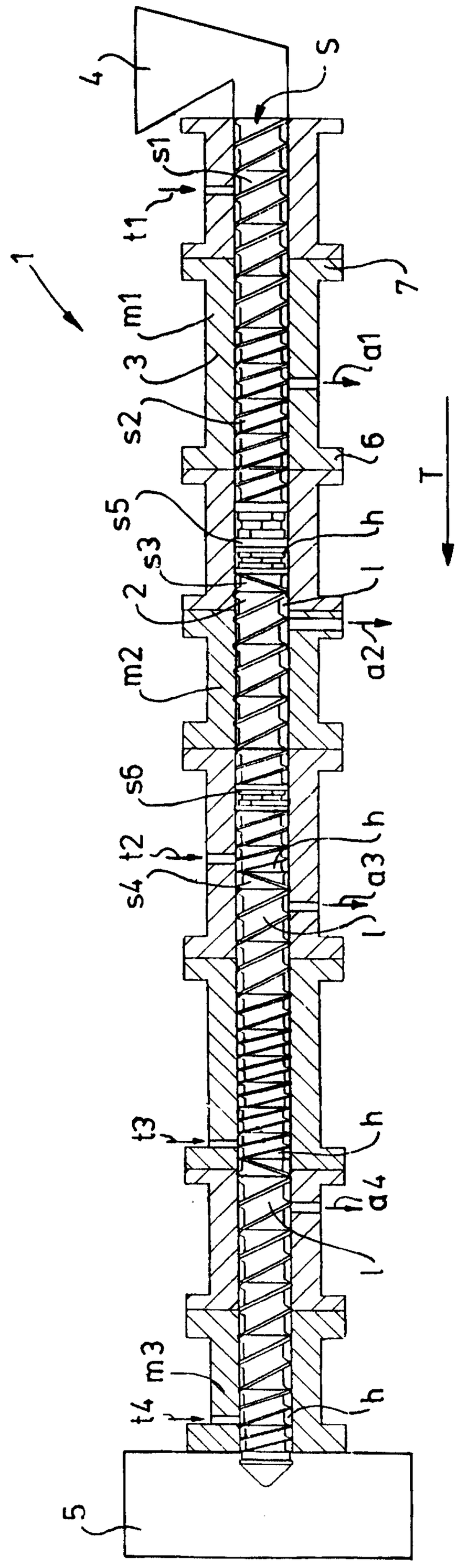
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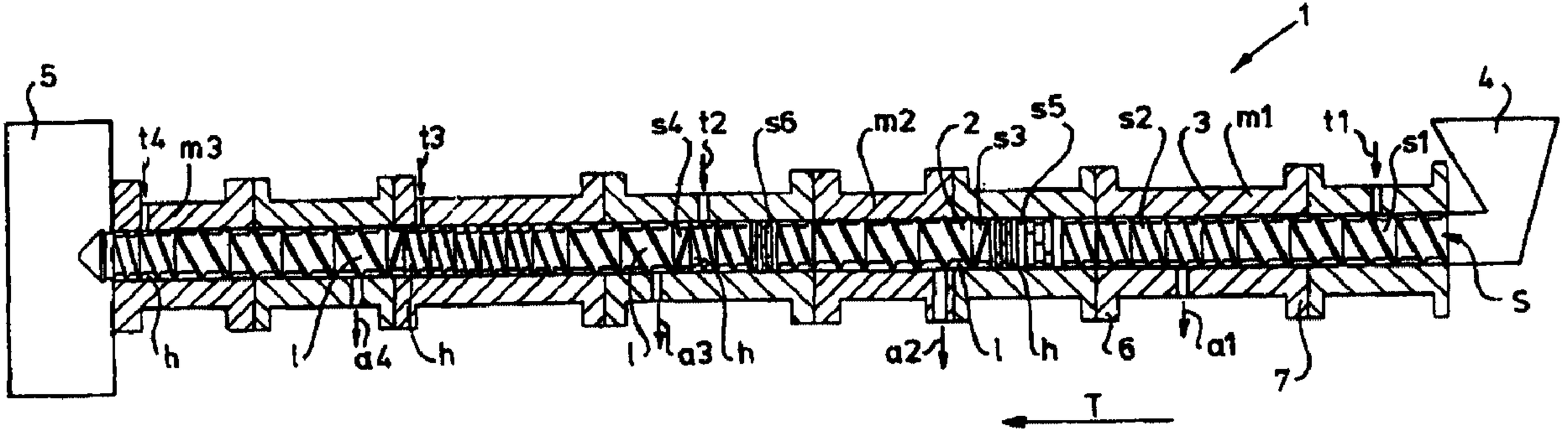
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