⑲ Europäisches Patentamt
European Patent Office
Office européen des brevets

⑪ Publication number : **0 199 481**
**B1**

## ⑫ EUROPEAN PATENT SPECIFICATION

㊺ Date of publication of patent specification : 13.12.89

㉑ Application number : 86302491.5

㉒ Date of filing : 04.04.86

(51) Int. Cl.$^4$ : D 21 C 3/26

# (54) A process for the manufacture of cellulose pulps.

㉚ Priority : 04.04.85 NZ 211684

㊸ Date of publication of application : 29.10.86 Bulletin 86/44

㊺ Publication of the grant of the patent : 13.12.89 Bulletin 89/50

(84) Designated contracting states :
DE FR GB SE

(56) References cited :
CA–A– 1 145 106
FR–A– 2 544 757
US–A– 4 502 918

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## Description

This invention relates to a process for the manufacturing of cellulose mechanical pulps.

There are many known processes for the manufacture of cellulose pulps and, more particularly, high-yield cellulose mechanical pulps. These processes include but are not confined to refiner mechanical processes (RMP), thermo-mechanical processes (TMP), chemi-mechanical processes (CMP), thermo-chemi mechanical process (TCMP) and chemi-thermo mechanical processes (CTMP). These processes which have been developed over the years are essentially developments of the basic RMP process and whilst the differences between processes appear to be small they do represent improvements which lead to the production of improved pulp quality (often but not always coupled with reduced pulp yield). Generally, however, most of the improved processes have been at the expense of an increase in refining energy required to meet the requisite paper making or other end use properties.

Mechanical pulp plants typically have a lower specific capital cost to build and mechanical pulps typically have a high yield and can be cheaper to produce than chemical pulps. In addition mechanical pulps do not provide the pollution problems which are associated with chemical pulps. Generally mechanical pulps have not been of the same high quality as chemical pulps and thus to achieve an end pulp which is of the required quality but at the same time economic to produce it is a well known technique to combine mechanical and chemical pulps. The aforementioned developments in mechanical pulp processes which stem from the basic RMP process have led to some mechanical pulps having the characteristics of a chemical pulp and they can thus be used as a replacement, in whole or in part, for chemical pulps in those applications where chemical pulps have traditionally been used.

For example, there is disclosed in Canadian patent specifications 1 071 805, 1 145 106 and 1 145 107 to Ontario Paper Company Ltd processes for the production of mechanical pulps which are claimed to be useful as substitutes for chemical pulps. These processes require a mechanical pulp of the TMP, RMP, or TCMP type to be treated or mixed with a relatively high charge of digestion chemicals e. g. an aqueous sodium sulphite solution after the refining stage and the thus treated pulp to be pressure digested with optionally further refining taking place after the digestion stage.

In U.S. patent specification 4 502 918 of MacMillan Bloedel Ltd there is disclosed a pulping process in which wood particles are treated with an aqueous solution of sodium sulphite, the treated particles are then digested and refined following which the resultant pulp is separated into an accepts fraction and a rejects or long fibre fraction. The rejects fraction is then further treated with a sodium sulphite solution, digested and refined where upon the or at least part of the accepts and rejects fractions are combined to form the final pulp.

The present invention has its basis in the surprising discovery that the whole pulp can be subjected to a two stage digestion and refining process with the second addition of an aqueous sodium sulphite solution taking place prior to or with the cellulose material entering or after the first refining stage followed by digestion after the chemical addition. The process can be carried out with significant savings in energy requirements even though the whole pulp is processed in the two digestion and refining steps whilst increase in many of the desirable properties of the pulp for end use applications are obtained. The process has particular application either singly or in combination for pinus radiata corewood and/or pinus radiata slab wood, eucalyptus and New Zealand hardwoods but it is believed that it has similar application to other wood types and species.

Whilst the present invention requires a relatively high total charge of digestion chemicals the total charge is split into two separate charges of which the first charge is preferably lower but preferably in an impregnation process rather than a simple mixing stage. The resultant pulp is low in fines, has high strength characteristics and is produced with low energy requirements. For example, the total chemical charge in the present invention may be in the order of that required in a CMP process but does not require the high energy demands of a CMP process for some wood types, particularly pine species and hardwood/softwood blends.

FR-A-2 544 757 describes a process of formation of pulp from fibre, such as bagasse and bamboo, involving at least two digestion stages, with addition of digestion chemicals before each stage. « Blowdown », i. e. decompression, occurs between two digestion stages, in order to separate the fibre bundles.

The object of the present invention is to provide a process for the manufacture of cellulose mechanical pulps which provides a high yield pulp of improved quality which can be used as a substitute for chemical pulps and is achieved without increases or at lesser increases in refining energy inputs.

The invention is set out in claim 1.

Broadly, the invention can be said to consist of a process for the manufacturing of cellulose pulp using two or more chemical addition and digestion stages in conjunction with mechanical pulping techniques.

The term « mechanical pulping » as used herein has its normal meaning in the art and refers to the process of disruption of a cellulose substance by mechanical action to yield a product consisting mainly of liberated and separated single cellulose fibres and their fragments and which is suitable for use in the manufacture of paper and other products.

More specifically the invention consists of a process for the manufacturing of a cellulose pulp from material in particulate form comprising the steps of impregnating the material with digestion chemicals, digesting the impregnated material, adding further digesting chemicals and mechanically refining the resultant mixture to reduce the material to predominantly whole fibres, digesting the impregnated and refined mixture and then further refining the resulting pulp product, if required.

The following is a more detailed description of an embodiment of the invention, with reference to the accompanying drawing which is a schematic flow diagram of a process of the invention.

According to the process shown in the drawing, wood chips are washed in washer 10 and conveyed to a surge/pre-steaming vessel 11. In this vessel live steam is added to remove air and preheat the chips to a temperature in the range of 80-100 °C. Discharge of chips from vessel 11 is maintained at a controlled rate to a compression screw feeder 12 wherein excess liquid is removed and the chips are dewatered to over 20 % dryness. In the preferred form the chips are dewatered to over 40 % dryness. The chips are then delivered to an impregnation device 13 (preferably an inclined screw conveyor).

Digestion chemicals are delivered to or between the feeder device 12 and/or the impregnation device 13 with delivery being at a temperature below 100 °C, preferably under 50 °C. These delignifying chemicals in aqueous form are of known composition, primarily of the sodium sulphite family, and are added at a level of 0,5 to 5 % active chemical on bone dried fibre. Condensation of steam within the chips causes the liquor to be absorbed into the chips. Retention time within the liquor zone of the impregnation device 13 is 5 to 20 minutes but preferably over 7 minutes.

On leaving the liquor zone of the impregnation device 13 the chips may be subjected to direct steam heating to raise the temperature of the chips to within the range of 80-100 °C. The impregnated chips are then fed to a primary digester 14 by a pressure sealing device 13a such as a compression screw, rotary valve or similar. The residence time within the digester 14 is in the range of 3 to 20 minutes, but preferably 5 to 10 minutes, with heating by direct steam injection to give a vapour phase reaction. The pressure of the primary digester 14 is in the range of 0-800 kPa overpressure, but more preferably 200 to 400 kPa overpressure.

Product from primary digester 14 is controlled by metering in accordance with known procedures and fed via feeder/pressure seal 15 to a primary refiner 16 (such as a disc refiner) operating at 0 to 600 kPa overpressure but more preferably 200 to 300 kPa overpressure. Further digestion or cooking chemicals of known composition are added to the product feed to the primary refiner 16 for subsequent reaction in the secondary digester 19. The cooking chemicals are mixed with the chip/pulp mixture present in the primary refiner 16 by the refiner action. Addition of the sulphite based cooking chemicals is in the range of 2 to 20 % active chemical on bone dried fibre preferably 5 % to 10 % on bone dried fibre.

The pulp and steam generated in the primary refiner 16 are discharged through a common blowline to a cyclone 17 where water and/or spent process liquor is added for consistency control during the subsequent digestion phase. Pulp and liquor is discharged from cyclone 17 by a feeder/pressure seal device 18 (preferably a compression screw) and enters the secondary digester 19. The residence time within the secondary digester 19 is in the range of 20 to 120 minutes but more preferably 30 to 60 minutes at an overpressure in the range of 100 to 800 kPa but more preferably 300 to 600 kPa overpressure.

Discharge from the secondary digester 19 is achieved by injecting water/spent liquor through lines 20 and 21 into the base of the secondary digester 19 and mixing this with the pulp. The motive force for discharging the pulp is provided by the internal pressure in the digester. Alternatively the digester may be discharged by screw conveyor or other methods.

Discharged pulp is passed to a washer 22, preferably a press washing system, wherein it is washed and pressed and passed by a feeder/pressure seal 23 to a secondary refiner 24. The secondary refiner 24 may operate at a pressure in the range 0 to 600 kPa overpressure but preferably 200 to 300 kPa overpressure. Steam and pulp are discharged from secondary refiner 24 through a common blowline to a cyclone 25 where they are separated and discharged in substantially the same manner as that from the primary refiner 16.

The resultant pulp may then be treated in accordance with accepted pulping practices, such as latency removal, screening, cleaning and bleaching. The resulting pulp product may be dried or used directly or indirectly for its intended end use.

The process as described above is open to modification. For example, either or both the refining stages in refiners 16 and 24 may take place at atmospheric pressure with screws or other discharge and conveying devices ; two or more stages of refining may be present prior to or after the secondary digester 19 ; pulp from the secondary digester 19 may be discharged directly from the pulping process outlined as being an inter- or post- refining stage ; inter-stage washing and de-watering could be included between first stage digesting and first stage refining ; a further stage or stages of chemical addition, refining, digesting with or without inter-stage washing and de-watering could be added.

Impregnation/cooking chemicals for the first digestion stage may be added via a mechanical mixing system, similarly for the second digestion stage they may be added to the cellulose material after the primary refiner in a mechanical mixing system.

The effect of the inter-stage or post-digesting stage is to give a change in fibre properties, improving some, but what is of significance, is a major reduction in refining energy requirements to achieve the desired end use properties.

The process according to the present invention results in a high-yield mechanical pulp of at least a similar but generally improved quality over pulps obtained with similar known process but without increases or at lesser increases in refining energy inputs. High levels of combined sulphur at the same freeness level can thus be achieved without higher power usage as is normally the case with some chemi-mechanical (CMP) or chemi-thermomechanical (CTMP) processes. The pulp is referred to in the following tables as sulphonated chemi-thermomechanical pulp (SCTMP).

The digestion/refining process treatment allows high sulphonation to be achieved giving better brightness, absorbency, softness and fibre flexibility with a major reduction in refining energy requirements. In addition the process results in clean fibre separation, improved fibre yield with reduced fines and improved bulk, tear index, tensile index, stretch, burst and other wet and dry web properties.

Test runs of the process of the present invention have been carried out and the results of the properties of the process and RMP and CTMP pulp for unbleached screened and cleaned pulp (tested to SCAN standards using a 60 g/m$^2$ handsheet) are set out below.

The pulp product prior to bleaching stage (s) contains a low quantity of fibre bundles, that is less than substantially 10 % and preferably substantially none.

The process can be used for soft woods, blends of soft woods and blends of softwoods and hardwoods. For the last case superior quality is obtained than when using other high yield processes.

Mesh sizes in the table below are measured according to the Bauer McNett method. Shive content is measured by the Sommerville method. All results in the table are test results for Pinus Radiata slab wood (sawmill residue) chips. All tests are carried out on 60 g/m$^2$ hand sheets according to ISO standards.

The following examples of the present invention and conventional chemi-thermomechanical pulp process (CTMP) and refiner mechanical pulp process (RMP) were carried out in a pilot plant using commonly available New Zealand wood species.

## Example 1 (comparative)

An RMP process was carried out on softwood chips (Pinus Radiata, sawmill slab residue) at about 60 % moisture content, the chips being refined with two passes through a double disc refiner at atmospheric pressure. The results are shown in the table in the column headed « RMP » and establish a base for comparison with other pulping processes.

## Example 2 (comparative)

A conventional CTMP process was carried out. Blends of chips of different wood species and single species (see first line in the following table) were treated in a steaming vessel for 10 minutes at about 95 degrees centigrade, condensate was drained and the vessel charged with cold impregnation chemical, sodium sulphite, of sufficient concentration to give the desired final sulphur content. The impregnation liquor was heated by recirculation through an external heat exchanger to about 95 degrees centigrade and the liquor then drained. The impregnated chips were digested at about 130 degrees centigrade using direct steam injection for 5 minutes. The digested chips were discharged by screw conveyor and fed to a pressurized double disc refiner operating at about 2 bar overpressure. The resulting pulp was again refined by a second pass through the same refiner under the same pressure conditions.

The results of the processes were listed under the columns CTMP in the following table.

## Example 3

A process according to the present invention was carried out. Blends of chips of different wood species and single species (see first line in the following table) were treated in a steaming vessel for 10 minutes at about 95 degrees centigrade, condensate was drained and the vessel charged with cold impregnation chemical, sodium sulphite, of sufficient concentration to give the desired final sulphur content. The impregnation liquor was heated by recirculation through an external heat exchanger to about 95 degrees centigrade and the liquor then drained. The impregnated chips were digested at about 130 degrees centigrade using direct steam injection for 5 minutes.

The digested chips were discharged by screw conveyor and fed to a pressurized double disc refiner. At the point of feeding the refiner secondary digestion chemical (sodium sulphite liquor) was added at the required concentration to give the desired final pulp sulphur content. The pulp/liquor mixture was conveyed by refiner generated steam (blown) back to the primary cooking vessel and digested at 160 degrees centigrade for about 60 minutes. Digested pulp was discharged and refined for a second time. Both refining stages were pressurised at about 2 bar overpressure.

The results of the processes are shown in columns (marked SCTMP) in the following table.

## Example 4

The process of Example 3 was carried out. The pulp had further digestion chemicals added at the entry to the second stage refiner and a third stage of digestion was carried out followed by a third stage of refining at atmospheric pressure. The results of this process are shown in the extreme right hand column marked « SCTMP » in the table which follows.

In the foregoing examples all final pulp was washed and screened through a 0.16mm flat screen prior to property testing to simulate the screening/cleaning function of a typical mechanical pulp mill.

The resultant pulp from the process according to the invention is unexpectedly suitable for further refining development to improve various properties, in particular tensile index, burst, air resistance, that will enhance its performance in its selected end use.

In its undeveloped and developed stage, it is suitable for such end uses as a chemical pulp substitute either directly or in part, in such products as fluffing pulp and various papers such as, but not limited to, tissue, mechanical printing grades, liner board and board.

| PULPING PROCESS | RMP | CTMP | SCTMP | CTMP | SCTMP | SCTMP | CTMP | SCTMP |
|---|---|---|---|---|---|---|---|---|
| Slab P. Radiata, % | 100 | 100 | 100 | | | 50 | 35 | 35 |
| Juvenile P. Radiata % | | | | 100 | 100 | 50 | 35 | 35 |
| E. Regnans, % | | | | | | | | 30 |
| Tawa, % | | | | | | | 30 | |
| Can. Std Freeness, ml | 169 | 389 | 386 | 562 | 435 | 289 | 325 | 235 |
| Tensile Index, Nm/g | 31.1 | 34.2 | 36.4 | 20.8 | 27.4 | 38.3 | 15.9 | 47.2 |
| Stretch, % | 2.1 | 1.1 | 2.6 | 1.5 | 2.4 | 2.8 | — | — |
| Tear Index, Nm m$^2$/g | 8.4 | 11.7 | 9.58 | 8.1 | 9.32 | 8.86 | 4.85 | 7.87 |
| Burst Index, kPa m$^2$/g | 1.69 | 1.74 | 2.11 | 1.04 | 1.42 | 2.25 | 0.78 | 3.14 |
| Sulphur Content, % | 0 | 0.51 | 0.56 | 0.41 | 0.40 | 0.72 | 0.12 | 0.59 |
| Absorption Capacity, g/g | 10.3 | 10.1 | 10.3 | 10.7 | 10.7 | 10.3 | — | — |
| DCM Extractives, % | 0.25 | 0.11 | 0.28 | 0.19 | 0.14 | 0.18 | 0.13 | 0.14 |
| Bulk, cm$^3$/g | 2.61 | 2.75 | 2.38 | 3.83 | 3.06 | 2.26 | 3.65 | 2.09 |
| Density, kg/m$^3$ | 383 | 364 | 420 | 261 | 327 | 442 | 274 | 478 |
| Brightness, % ISO | 52.4 | 49.7 | 53.2 | 54.8 | 57.5 | 51.6 | 47.9 | 53.8 |
| B. McNett 297 µm (48 mesh), % | 61.1 | 73.3 | 71.2 | 79.6 | 70.6 | 68.1 | 55.3 | 65.6 |
| B. McNett 74 µm (200 mesh), % | 25.2 | 19.0 | 18.8 | 9.3 | 17.9 | 20.6 | 21.2 | 18.4 |
| Sommerville Shive, % | 0.25 | 0.12 | 0.10 | 0.04 | 0.02 | 0.05 | 0.80 | 0.07 |
| PFI Minishive, % | 0.45 | 0.30 | 0.20 | 0.35 | 0.10 | 0.10 | 0.07 | 0.25 |
| Total specific refining energy, MWh/tonne (100) | 1.70 | 2.27 | 1.45 | 2.07 | 2.22 | 1.75 | 2.05 | 2.07 |

Notes :

1) Slab P. Radiata : Pinus Radiata sawmill residue ; Juvenile P. Radiata : Pinus Radiata plantation thinnings under 15 years : E. Regnans : Eucalyptus Regnans plantation grown hardwood ; Tawa : N.Z. native hardwood.

2) All tests carried out to ISO standards on 60 g/m$^2$ handsheets.

## Claims

1. A process for the manufacture of cellulose pulp from wood material in particulate form comprising the steps of
   (a) adding digestion chemicals to the material,
   (b) after step (a), digesting the material,
   (c) after step (b), adding further digesting chemicals to the digested material mixture, and
   (d) after step (c), further digesting the mixture

characterised in that, before, simultaneously with or after step (c), the mixture is mechanically refined to reduce the material to predominantly whole fibres so that step (d) is performed on a refined mixture.

2. The process according to Claim 1 wherein the pulp product resulting from digesting step (d) is further refined by mechanical action.

3. The process according to Claim 1 or Claim 2 wherein in step (a) digestion chemicals are added to the particulate material so as to impregnate it at a temperature of below 100 °C, with the amount of active chemical being in the range of about 0.5 to 5 % based on bone dried fibre.

4. The process according to Claim 3 wherein the impregnation of the particulate material takes place over a time period of about 5 to 20 minutes.

5. The process according to Claim 3 or Claim 4 wherein the impregnated material is fed, at a temperature in the range of about 80-100 °C, to a first digester wherein it is heated by direct steam at an overpressure in the range of about 0-800 kPa for a period of about 5 to 10 minutes to perform step (b).

6. The process according to Claim 5 wherein the mixture issuing from the first digester is refined in a primary refiner with further digestion chemicals added to the said mixture from the digester (step (c)), said digestion chemicals being sulphite-based with the amount cf active chemical therein being in the range of about 2 to 20 % on bone dried fibre.

7. The process according to Claim 6 wherein the further digestion chemicals are added (step (c)) prior to the mixture being refined in the primary refiner.

8. The processing according to Claim 6 wherein the further digestion chemicals are added (step (c)) to the mixture within the primary refiner.

9. The process according to any one of Claims 6, 7 and 8 wherein the pulp and liquor from the primary refiner is digested (step (d)) at an overpressure in the range of about 100 to 800 kPa for a period of time in the range of about 30 to 60 minutes.

## Patentansprüche

1. Verfahren zur Herstellung von Cellulosepulpe aus teilchenförmigem Holzmaterial, das die folgenden Stufen umfaßt:

(a) Zugabe von Digerierchemikalien zu dem Material,

(b) Digerieren des Materials nach der Stufe (a),

(c) Zugabe von weiteren Digerier-Chemikalien nach der Stufe (b) zu der digerierten Materialmischung und

(d) weiteres Digerieren der Mischung nach der Stufe (c),

dadurch gekennzeichnet, daß vor, gleichzeitig mit oder nach der Stufe (c) die Mischung mechanisch raffiniert wird, um das Material zu überwiegend ganzen Fasern zu zerkleinern, so daß die Stufe (b) mit einer raffinierten Mischung durchgeführt wird.

2. Verfahren nach Anspruch 1, worin das aus der Digerierstufe (d) resultierende Pulpenprodukt auf mechanischem Wege weiter raffiniert wird.

3. Verfahren nach Anspruch 1 oder 2, worin in der Stufe (a) Digerierchemikalien dem teilchenförmigen Material bei einer Temperatur unter 100 °C zugesetzt werden, um es damit zu imprägnieren, wobei die Menge der aktiven Chemikalie in dem Bereich von etwa 0,5 bis 5 %, bezogen auf die staubtrockenen Fasern, liegt.

4. Verfahren nach Anspruch 3, worin die Imprägnierung des teilchenförmigen Materials über einen Zeitraum von etwa 5 bis 20 Minuten durchgeführt wird.

5. Verfahren nach Anspruch 3 oder 4, worin das imprägnerte Material bei einer Temperatur in dem Bereich von etwa 80 bis 100 °C einem ersten Digestor zugeführt wird, in dem es zur Durchführung der Stufe (b) durch Direkt-Wasserdampf unter einem Überdruck in dem Bereich von etwa 0 bis 800 kPa für eine Zeitspanne von etwa 5 bis 10 Minuten erhitzt wird.

6. Verfahren nach Anspruch 5, worin die aus dem ersten Digestor austretende Mischung in einer primären Raffiniereinrichtung raffiniert wird, wobei weitere Digerierchemikalien der aus dem Digestor austretenden Mischung zugesetzt werden (Stufe (c)), wobei es sich bei den Digerierchemikalien um solche auf Sulfit-Basis handelt und die Menge der aktiven Chemikalie darin in dem Bereich von etwa 2 bis 20 %, bezogen auf die staubtrockenen Fasern, liegt.

7. Verfahren nach Anspruch 6, worin die weiteren Digerierchemikalien zugegeben werden (Stufe c)), bevor die Mischung in der primären Raffiniereinrichtung raffiniert wird.

8. Verfahren nach Anspruch 6, worin die weiteren Digerierchemikalien der Mischung innerhalb der primären Raffiniereinrichtung zugesetzt werden (Stufe c)).

9. Verfahren nach einem der Ansprüche 6, 7 und 8, worin die Pulpe und der Liquor aus der primären Raffiniereinrichtung digeriert werden (Stufe (d)) unter einem Überdruck in dem Bereich von etwa 100 bis 800 kPa für eine Zeitspanne in dem Bereich von etwa 30 bis 60 Minuten.

## Revendications

1. Procédé de fabrication de pâte cellulosique à partir de matériau de bois sous une forme particulaire comprenant les étapes de :

(a) ajouter des produits chimiques de digestion au matériau,

(b) après l'étape (a), digérer le matériau,

(c) après l'étape (b) ajouter d'autres produits chimiques de digestion au mélange digéré, et

(d) après l'étape (c) digérer encore le mélange

caractérisé en ce que, avant, simultanément avec ou après l'étape (c), le mélange est mécaniquement raffiné pour réduire la matière de manière prédominante à des fibres entières de façon que l'étape (b) soit accomplie sur un mélange raffiné.

2. Procédé selon la revendication 1 où la pâte produite résultant de l'étape de digestion (d) est de plus raffinée par action mécanique.

3. Procédé selon la revendication 1 ou la revendication 2 où à l'étape (a) des produits chimiques de digestion sont ajoutés à la matière particulaire afin de l'imprégner à une température inférieure à 100 °C, la quantité du produit chimique actif étant comprise entre environ 0,5 et 5 % en se basant sur la fibre archisèche.

4. Procédé selon la revendication 3 où l'imprégnation de la matière particulaire a lieu sur une période de temps d'environ 5 à 20 minutes.

5. Procédé selon la revendication 3 ou la revendication 4 où la matière imprégnée est introduite, à

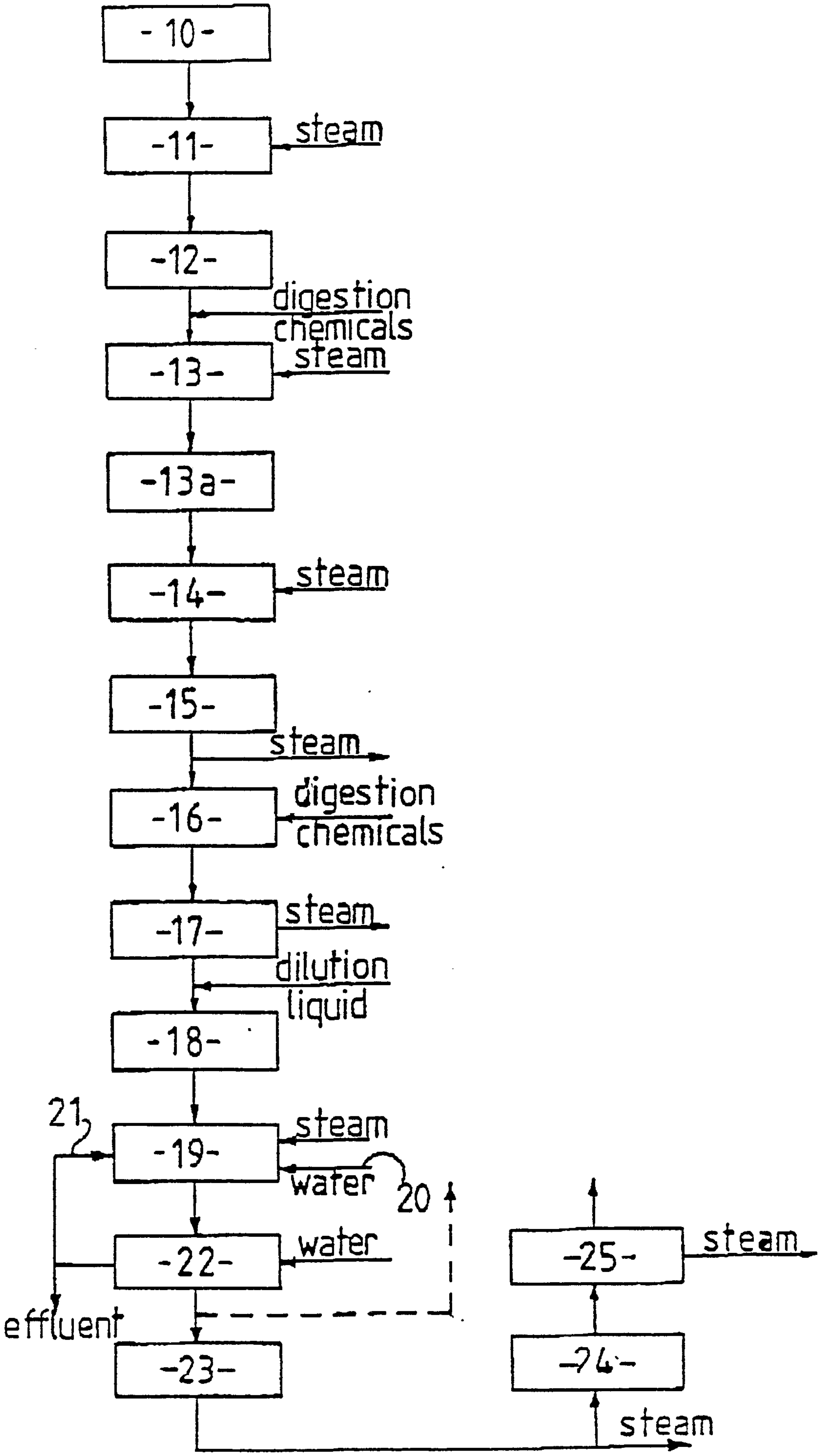
une température comprise entre environ 80 et 100 °C, dans un premier digesteur où elle est chauffée par vapeur directe à une surpression comprise entre environ 0 et 800 kPa pendant environ 5 à 10 minutes pour accomplir l'étape (b).

6. Procédé selon la revendication 5 où le mélange sortant du premier digesteur est raffiné dans un raffineur primaire avec d'autres produits chimiques de digestion ajoutés dans ledit mélange du digesteur (étape (c)), lesdits produits chimiques de digestion étant à base de sulfite avec la quantité de produits chimiques actifs comprise entre environ 2 et 20 % sur la fibre archisèche.

7. Procédé selon la revendication 6 où les autres produits chimiques de digestion sont ajoutés (étape (c)) avant que le mélange ne soit raffiné dans le raffineur primaire.

8. Traitement selon la revendication 6 où les autres produits chimiques de digestion sont ajoutés (étape (c)) dans le mélange à l'intérieur du raffineur primaire.

9. Procédé selon l'une quelconque des revendications 6, 7 ou 8 où la pâte et la liqueur du raffineur primaire est digérée (étape (d)) à une surpression comprise entre environ 100 et 800 kPa pendant une période de temps comprise entre environ 30 et 60 minutes.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