PUBLISHED

MAY 4, 1943.

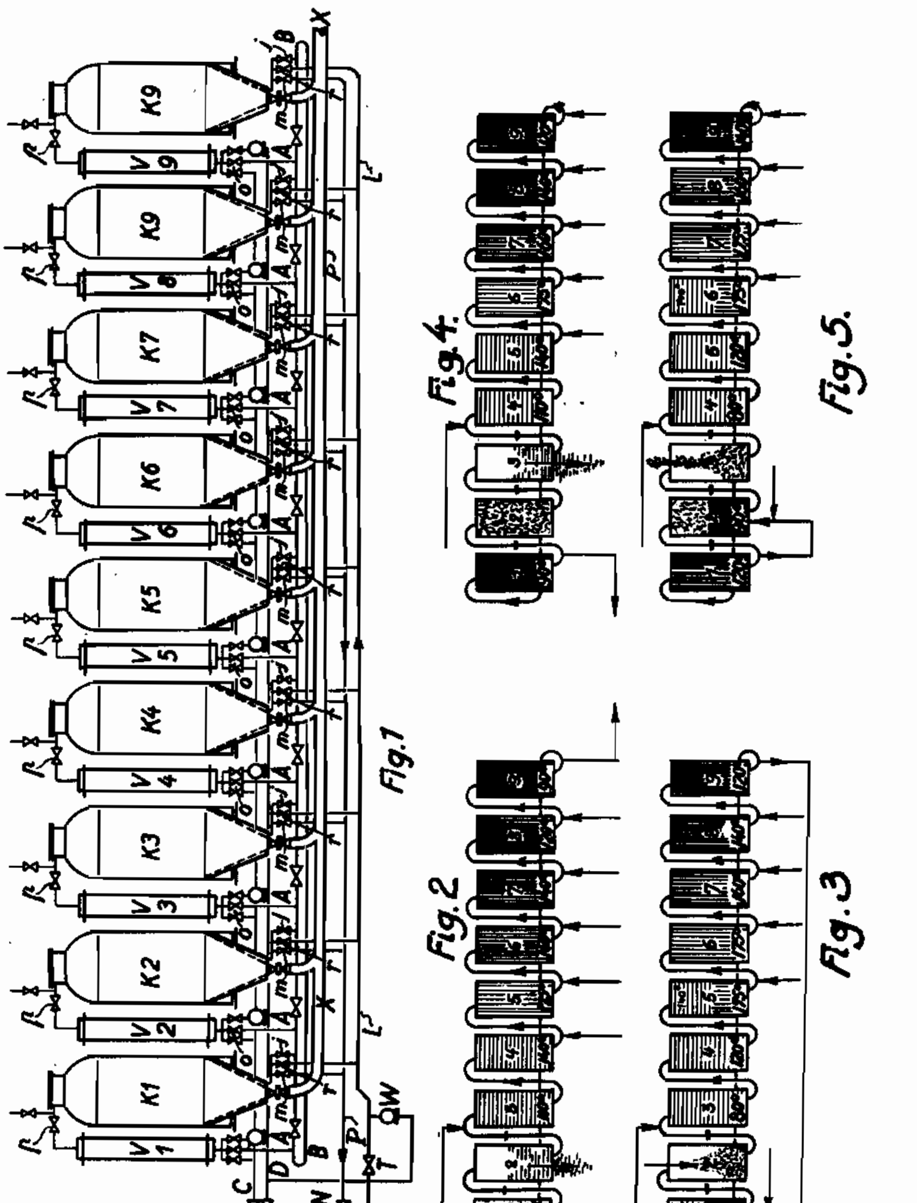
BY A. P. C.

R. JENKE

PROCESS AND APPARATUS FOR TREATING OR IMPROVING CELLULOSE MATERIAL

Filed Dec. 27, 1938

Serial No. 247,889



Inventor

Richard Jenke

By Rudolf Hilfsmann

his Attorney

Published May 4, 1943 **Serial No. 247,889**

ALIEN PROPERTY CUSTODIAN

PROCESS AND APPARATUS FOR TREATING OR IMPROVING CELLULOSE MATERIAL

Richard Jenke, Saalfeld, Germany; vested in the Alien Property Custodian

Application filed December 27, 1938

The invention relates to a process and apparatus for treating or improving cellulose material and enables a greater yield to be obtained of a material which is specially suitable for the production of viscose and artificial silk.

The process according to the invention consists in that the material is maintained at a definite temperature and an adjustable pressure independent of the temperature and is treated in a plurality of successive stages with lyes containing only small quantities of caustic alkali or with water for washing, the water and/or the lye flowing continuously through the material until the lye has acquired the desired concentration of salts, alkali solution preferably in concentrated form being added between each stage to bring the lye to the concentration necessary for treating the raw material.

The lye is allowed to flow through the material until it becomes so rich in salts that their recovery becomes economical. The raw material is treated with lye of maximum strength and the finished or nearly finished material with weak lye or water. In order to obtain the best effect, the material is treated at each stage with lye of only slight causticity while the total causticity used for the complete treatment has the usual value.

The lye concentration is kept below a value considerably influencing the viscosity of the cellulose solution to be obtained. The causticity of the strongest lye solution is almost completely utilised and the solution is rapidly removed from the material and can therefore not affect its bleaching capacity. The material is washed in the absence of air, oxygen or carbon dioxide since it is not transferred from the boiler to a separate washing plant, thus avoiding heat losses and chemical or mechanical damage due to rubbing on the walls of pipes, and destruction of the fibres by sudden expansion after the release of pressure.

The process according to the invention is described hereinunder by way of example with reference to the accompanying drawings but it is to be understood that the invention is not limited to the particular details set forth therein. In said drawings:

Fig. 1 is a general diagrammatic view of a plant according to the invention;

Figs. 2 to 5 represent diagrammatically various stages of the process effected by means of the plant of Fig. 1.

The plant shown in Fig. 1 comprises a set of nine boilers K1—K9, each boiler being provided with a pre-heater V_1—V_9 for heating the water or lye flowing therein to the best temperature for the particular stage of the material being treated.

Each boiler further comprises supply means A for adding alkali lye in adjustable quantities to the water or lye from a common pipe D, connections for the lye, washing water and waste water piping to a common ring piping B, and a pipe C which is connected to a washing water supply E fed by a pump F and which is connected to conduits L and P. At the end of the latter is located an automatic regulating valve N by means of which concentrated lye flowing in pipe P can be conveyed either to the regenerating station or through pipe L to any desired boiler.

The pressure of the water supply may be regulated by means of a compressor so that it is always slightly greater than the pressure in the boilers. Dosage pumps are advantageous for use as supply means A.

Fig. 2 represents one stage of the process at which the material is being distintegrated in five boilers 5—9, and boilers 3 and 4 are used used for washing. The boilers 3—9 are connected together through valves *j*, o, *p* and preheaters V (Fig. 1), and are traversed by washing water for boilers 3 and 4, and by lye for boilers 5—9. The water for boiler 3 flows from pipe C through its corresponding preheater V_3 and alkali is added through supply means A to each boiler 5—9, the concentrated lye flowing from the last boiler 9 through valve *m*, pipe P, and valve N to the regenerating station. The quantity of lye leaving boiler 9 is of course equal to the quantity of water which enters boiler 3 and the pressure of supply E thus governs the whole system.

The partly or completely treated material in boilers 5—9 is denoted by vertical hatching, the material in boiler 5 being almost completely disintegrated and that in boiler 9 being at the first stage of treatment, the intermediate boilers 6, 7, 8 being at intermediate stages. Boilers 3 and 4 contain finished material and are supplied with washing water only but after boiler 4 alkali is added through supply means A in an amount sufficient for completing the last stage of the treatment of the material in boiler 5. Similarly alkali is added between each of the following boilers in amounts sufficient to complete the appropriate stage of treatment.

The lye gradually increases in concentration of salts from boiler to boiler and is of maximum concentration or density when leaving boiler 9. The gradually decreasing disintegration stages of the material and increasing lye concentration from boiler 5 to boiler 9 is represented by different thicknesses of vertical hatching. When the material in boiler 2 is completely boiled and washed it is removed by pipe X. Boiler 1 is filled with raw material.

When the lye in boiler 9 is of sufficient strength it is removed through valve N until its density falls below a certain value dependent on the amount of dissolved salts, when it is again treated with alkali and led to boiler 1 (Fig. 3), for treating a fresh batch of material, the lye flowing from boiler 9 through valve m, pipe P, valve N and thence to boiler 1 through valve T, pipe L, valve r. The addition of alkali to the lye is effected through a cock W by means of which an exactly predetermined amount of alkali can be conveyed from pipe D to pipe L.

The quantity of lye removed from boiler 9 is replaced by an equal amount of fresh lye at boiler 5 (Fig. 2). In Fig. 3 the material in boiler 5 is completely treated and the addition of alkali between boilers 4 and 5 is stopped, only washing water being now supplied to boiler 5. When boiler 1 is filled with concentrated lye, boiler 5 is supplied with water and the lye in boiler 1 takes salts from the raw material therein and will be ready for regeneration.

The next stage of the process is represented by Fig. 4 which shows a system similar to Fig. 2 except that it has been displaced by one boiler towards the right-hand side. The material in boiler 3 is washed and removed through pipe X and boiler 2 is filled with a fresh batch of raw material. The treatment then continues as described with reference to Fig. 2, boiler 2 being supplied eventually with concentrated lye from boiler 1 as shown in Fig. 5 which represents a stage similar to Fig. 3 but displaced by one boiler to the right.

The process is then continued, the system following the sequence of Figs. 2, 3, or Figs. 4, 5, this sequence being repeated five times until the material in boiler 9 (Fig. 2) has reached the completed stage of the material in boiler 5 (Fig. 2). In the stages corresponding to Figs. 2 and 4 the concentrated lye flows to the regenerating station and a boiler containing the finished material is disconnected and emptied. In the stages corresponding to Figs. 3 and 5 a boiler filled with a batch of new material is put in the circuit, filled with lye and the supply of alkali is cut off from the boiler containing the finished material, the amount of concentrated lye drawn off being replaced each time by the addition of fresh lye.

The time required for the treatment of Figs. 2 and 4, or Figs. 3 and 5 depends on the material treated and the number of boilers. It may be calculated as follows:

If the material is to be completely treated in 5 hours and five boilers are used for successive stages of treatment, one stage, i. e. from the stage of Fig. 1 to Fig. 3, requires one hour. During this time the steps represented by Figs. 1 and 2 must be effected as well as boiler filling and lye removal. If the boiler capacity is 40 cubic metres and the lye removal 20 cubic metres, this gives a total of 60 cubic metres or 1 cubic metre/min. Lye filling of 40 cubic metres thus takes 40 minutes and the lye removal 20 minutes. The total boiling time is 5 hours and the washing time 2 hours if two boilers are used for washing.

If it is desired to complete the boiling in 4 hours and to use 6 boilers, each stage takes 40 minutes. For a boiler capacity of 40 cubic metres and lye removal of 20 cubic metres, the boiler filling takes

$$\frac{40 \times 40}{60} = 26.6 \text{ mins.}$$

and the lye removal 13.3 mins.

If according to the usual known alkali processes, raw material is treated, bleached and dissolved in an ammonia solution of copper oxide, the viscosity is at most 50, whereas with the process according to the present invention viscosities of 500–1,000 are obtained. Wood, for example pine or deal when treated by the process of the invention gives a viscosity of 200 and more with a maximum bleaching capacity. The chlorine necessary for obtaining a high whiteness value amounts at most to 1–2% of the dry material.

The process of the invention is also more profitable than the hitherto known methods. For pine or deal a yield of about 40% can be obtained whereas the usual process with a chlorine consumption of 2% gives a yield of only 35%. The strength of the material is also increased.

In addition to disintegrating raw material the process is also suitable for improving cellulose material. Hitherto for example sulphite material has been treated with a weak caustic soda solution, boiled and washed or subjected to similar after treatment. This requires large quantities of water which hold only small quantities of soda and salts the recovery of which is uneconomical. The quantity of sodium salt is, however, so great that the spent lye cannot be discharged into a river or canal. On the other hand according to the invention by using a series of boilers the lye is made so rich in sodium salts that the economical recovery of these salts is rendered possible.

RICHARD JENKE.