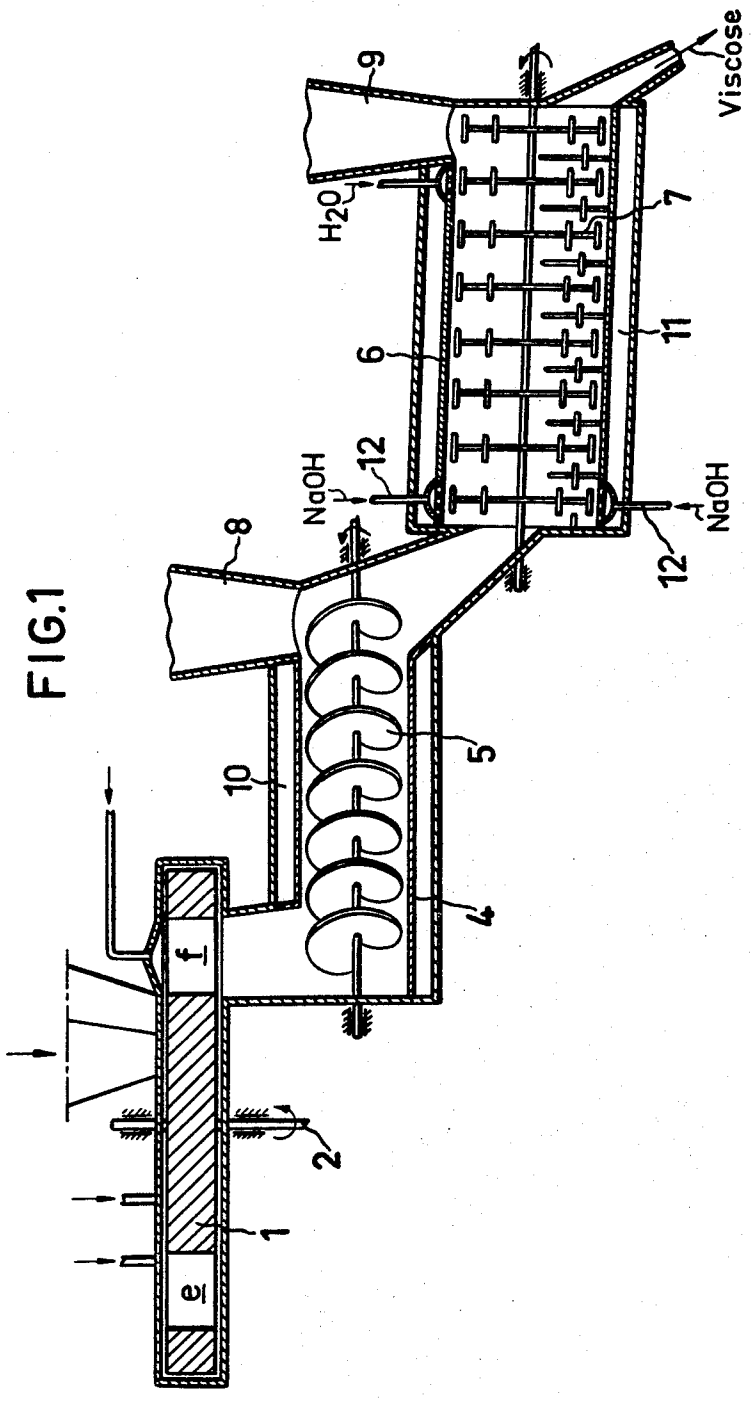
May 28, 1968 E. E. TREIBER 3,385,845

PROCESS AND DEVICE FOR THE XANTHATION OF ALKALI CELLULOSE

Filed April 5, 1963 2 Sheets-Sheet 1

FIG.1

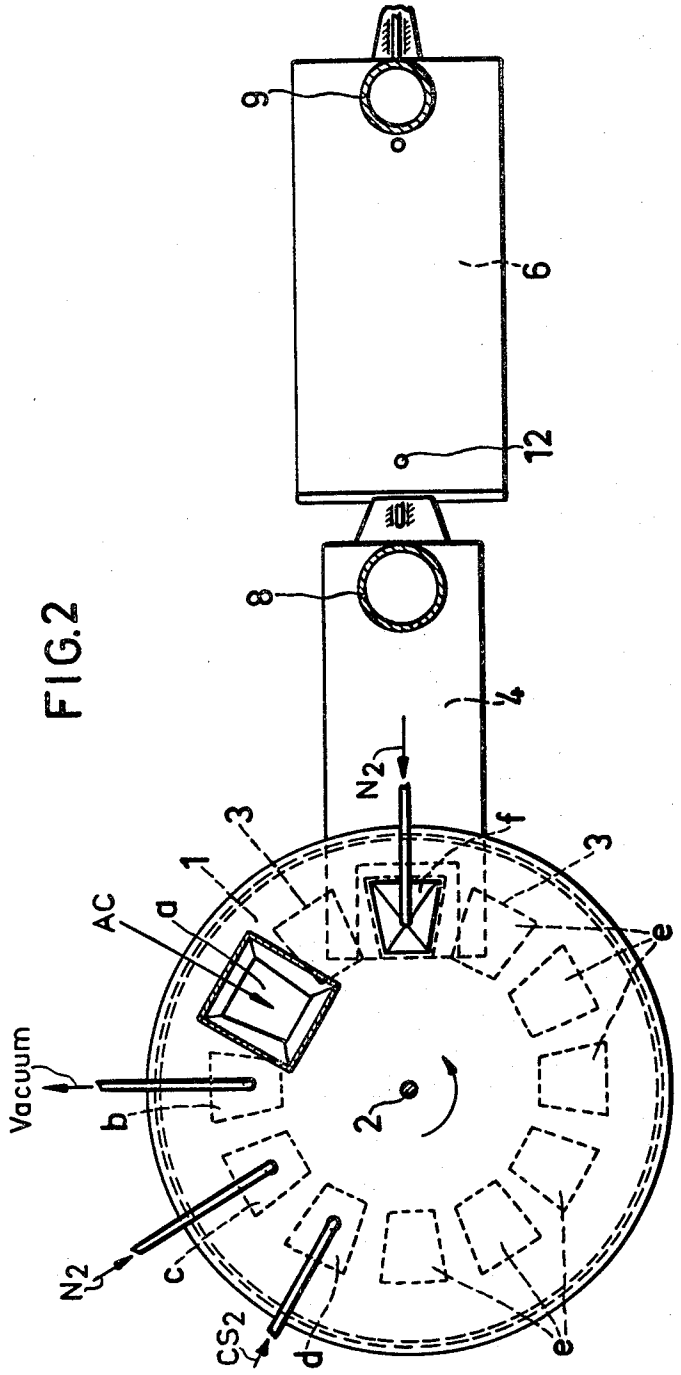


May 28, 1968 E. E. TREIBER 3,385,845

PROCESS AND DEVICE FOR THE XANTHATION OF ALKALI CELLULOSE

Filed April 5, 1963 2 Sheets-Sheet 2

FIG.2



United States Patent Office 3,385,845 Patented May 28, 1968

3,385,845

PROCESS AND DEVICE FOR THE XANTHATION OF ALKALI CELLULOSE

Erich Emmerich Treiber, Stockholm, Sweden, assignor to Sunds Verkstäder Aktiebolag, Sundsbruk, Sweden, a joint-stock company of Sweden

Filed Apr. 5, 1963, Ser. No. 271,024
Claims priority, application Sweden, Apr. 19, 1962, 4,454/62
2 Claims. (Cl. 260—217)

As is known, the viscose process and the spinning of fibres of regenerated cellulose can be carried out continuously and, thus, rationally except for the xanthation step. This continuous process is more and more used because it is more profitable and saves labour, and it is at present generally accepted for the production of viscose staple fibres and films. In order to reduce the interruption of the continuous process caused by the discontinuous xanthation step a number of apparatus have been constructed which enable a partial mechanization, epsecially as regards the emptying. Emptying by pumping off the more or less dissolved xanthate is an advantage also from the viewpoint of production, because thereby a xanthation in the gaseous phase is coupled together with a subsequent wet xanthation (emulsion xanthation), by which the xanthation passes from a heterogeneous reaction to a more or less homogeneous reaction. This result in a better state of solution of the viscose yielding advantages as regards the filtration and spinning. A drawback is the cleaning and drying of the apparatus after the reaction has been carried out. In spite of high automatization, however, no continuous xanthation is made possible by means of said apparatus.

Various proposals have been made for realizing a continuous viscose process. The processes hitherto known may be divided into two main groups.

One type of processes consists in carrying out the xanthation completely as a wet xanthation, mercerization and xanthation being in several cases carried out in one apparatus unit. In order thereby to obtain viscose with a good state of solution it is necessary to use greater charges of chemicals than in the conventional process, whereby the economical advantages are lost again, and a viscose is obtained which is not optimally adapted, nor can be adapted optimally with respect to the spinning or film casting.

The other type of processes refers to attempts to obtain a continuous churn. However, it has appeared that the carbon bisulphide consumption in this case becomes noticeably greater than in the conventional discontinuous process, chiefly due to leakage at the inlet and outlet.

The present invention is based on the conception that only the discontinuous method leads to a less expensive and more perfect solution of the problem and enables the production of all kinds of viscose, and that the discontinuous method hitherto used must be transformed into a continuous, i.e. a continuously discontinuous method.

Thus, the invention relates to a process for the xanthation of alkali cellulose in the manufacture of viscose in a continuous working operation, which comprises charging successively step by step each one of a number of cells with alkali cellulose, evacuating the cells, introducing protective gas, adding carbon bisulphide, continuously advancing the reaction mass from each cell into a following vessel for xanthation and, if desired, continuously advancing the contents from said vessel to a further vessel to provide wet xanthation by adding caustic soda solution and water. The reaction mass can be advanced by means of an indifferent solvent for carbon bisulphide, by which the introduction of carbon bisulphide into the reaction mass takes place simultaneously. Aliphatic or aromatic hydrocarbons, e.g. benzene or petroleum ether,

may be used as solvent for carbon bisulphide. This solvent is preferably added in the main reaction vessel and can, after finished reaction, be distilled off or separated by phase separation. The solvent can be used simultaneously with or without mechanical transport means. An adhesion of the reaction product may be counteracted by coating with polytetrafluoroethylene.

A suitable device for carrying out the process comprises a number of cells to be advanced successively step by step for charging with alkali cellulose in small portions, evacuation, introduction of protective gas and emptying, a main reaction vessel conected to the emptying cell for continuously advancing the reaction mass, said vessel being provided with homogenizing means and a vessel provided with agitating means for wet xanthation of the uncompletely xanthated alkali cellulose by addition of caustic soda solution and water. The cells may be arranged in a horizontal or vertical cell wheel and several such cell wheels may be connected in parallel. The main reaction vessel connected to the emptying cell and the wet xanthation vessel are provided with a thermostat jacket. The wet xanthation vessel is provided with a stirring member. The main reaction vessel and the wet xanthation vessel are provided with explosion protection chimneys.

An embodiment of a device according to the invention is illustrated schematically in the accompanying drawing in which FIG. 1 shows the device in longitudinal section and FIG. 2 is a horizontal view of the same.

The device consists of a horizontal cell wheel **1** which is mounted around a shaft **2**. The cell wheel **1** is provided with a number of cells **3** and to one of the cells there is connected a vessel **4** with a transport screw **5**, and the vessel **4** is connected to a wet xanthation vessel **6** which is provided with stirring members **7**. In both these vessels there are arranged explosion protection chimneys **8** and **9** respectively. The vessels **4** and **6** are provided with thermostat jackets **10** and **11** respectively.

The cell wheel is rotated stepwise in the direction of the arrow and the cells **3** are filled step by step with alkali cellulose (AC), (position *a*), are evacuated (position *b*), supplied with protective gas in the form of nitrogen gas (position *c*) and carbon bisulphide (position *d*). After a predetermined holding period (the positions *e*) the cell **3** is emptied when it reaches the position *f* with help of nitrogen gas, into the main reaction vessel **4** where the xanthation reaction continues and where unevenness, if any, is equalized. The xanthate formed is advanced slowly through the main reaction vessel **4** by means of the transport screw **5** and is introduced into the wet xanthation vessel **6**, where cooled soda lye is supplied through supply devices **12**. In the vessel **6** wet xanthation and dissolving of the xanthate take place. This wet xanthation vessel is preferably segmented and the stirring member **7** work up the mixture to a suitable homogeneity. At the outlet from the wet xanthation vessel **6** water may be added, if necessary. The viscose thus produced is drawn off continuously and can be conveyed to continuous dissolvers in the usual manner.

The cells emptied in the position *f* are immediately ready for receiving a new charge of alkali cellulose. The cells need not be washed or cleaned before receiving a new charge.

The invention is illustrated more in detail with reference to the following examples.

Example 1

To the alkali cellulose in a cell in the device described above there is added from between 25 to 65% of CS_2, calculated on the cellulose content of the alkali cellulose, after the cell has been evacuated and supplied with protective gas in the manner indicated above. The reaction

mass in the cell and in the following main reaction vessel is held at a temperature of 22–36° C. for 20 to 80 minutes. In the wet xanthation vessel there are added 2 to 5% soda lye, preferably of low temperature in such a quantity that, by the addition of water, finally a viscose with normal composition (5–10% of cellulose, 2.5–10% of NaOH) is obtained.

Example 2

Alkali cellulose from a continuous ageing equipment or mercerizing device is charged directly into the cells without special cooling so that the initial temperature at the xanthation will lie between 25 and 40° C. due to the hot alkali cellulose. In the main reaction vessel the reaction temperature is held at a lower value, e.g. at 23° C.

When carrying out the present method, a small amount of carbon bisulphide together with the dissolving lye may be added in the wet xanthation vessel. The dissolving in wet xanthation vessel may also be made only with water in order to obtain viscoses poor in chemicals.

What I claim is:

1. A process for xanthogenating alkali cellulose continuously step by step in the manufacture of viscose, which comprises introducing alkali cellulose into a compartment; removing air by evacuation from the alkali cellulose present in non-compressed dry condition in said compartment in a first step; introducing the alkali cellulose still in non-compressed dry condition together with carbon bisulphide into a second compartment in a second step while maintaining such conditions of pressure, time and temperature that the xanthation is carried out in the gaseous phase to a substantial degree but not completely; discharging the resulting reaction mass still in non-compressed condition to a following vessel; and completing the reaction in a third step by wet xanthation in the presence of caustic soda lye.

2. A process as claimed in claim **1**, in which the reaction mass is advanced with the aid of an inert hydrocarbon solvent for carbon bisulphide, said solvent being used simultaneously for introducing the carbon bisulphide into the reaction mass.

References Cited

UNITED STATES PATENTS

2,179,379	4/1965	Zenz et al.	259—4
2,508,987	12/1946	Booth	259—4
2,542,492	2/1951	Entwistle et al.	260—217
2,648,661	8/1953	Vandenburgh et al.	260—217
2,985,647	5/1961	Kohorn	260—217

FOREIGN PATENTS

488,761	8/1937	Great Britain.

DONALD E. CZAJA, *Primary Examiner.*

LEON J. BERCOVITZ, *Examiner.*

R. W. MULCAHY, *Assistant Examiner.*