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PROCESS FOR THE DESULFATION OF COAGULATING BATHS FOR THE SPINNING OF VISCOSE

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Fig 1

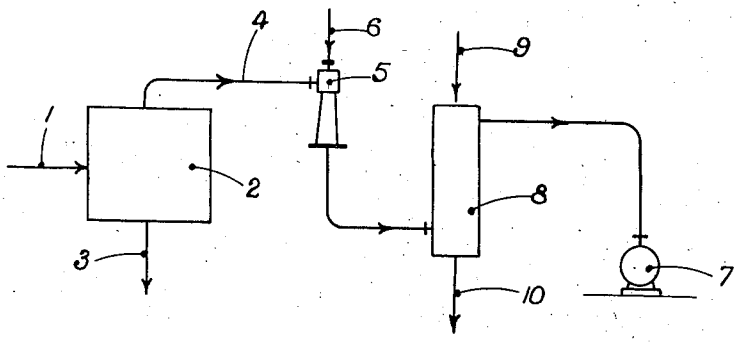
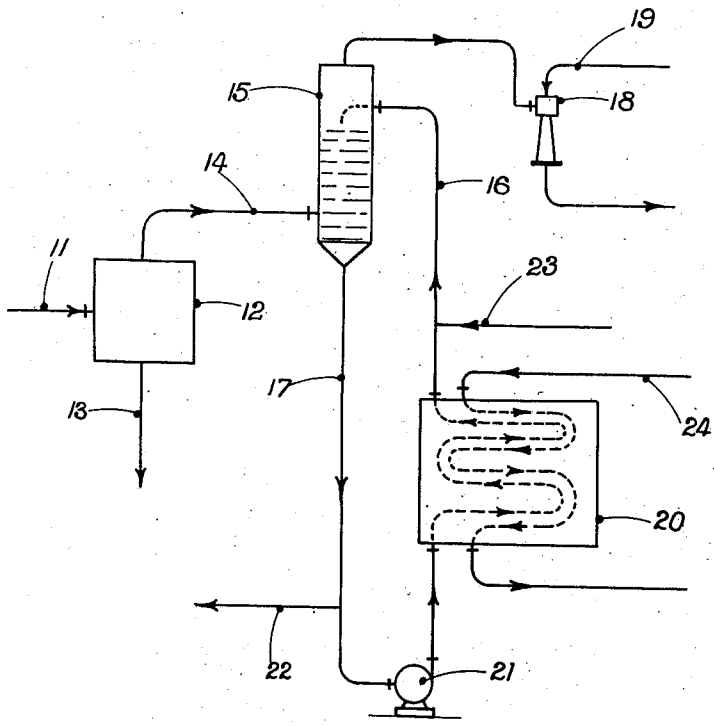


Fig 2



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PROCESS FOR THE DESULFATION OF COAGULATION BATHS FOR THE SPINNING OF VISCOSE

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2 Claims. (Cl. 18—54)

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This invention relates to a process for the desulphation of coagulation baths for the spinning of viscose.

It is known that in mills producing artificial textiles (rayon, fibranne) employing the viscose process, the coagulation is obtained by the passage of the thread through a bath of sulphuric acid, which enriches this bath in sodium sulphate, a product the excess of which must be eliminated.

This elimination is carried into effect by causing the sodium sulphate to crystallize by the cooling of a part of the bath in circulation in the manufacturing process.

According to the content in sodium sulphate which can be tolerated in the bath, content which varies according to the product which it is desired to obtain, it is necessary to treat, in the separation installation, a bath more or less rich in sodium sulphate. Besides this salt it will be appreciated that the other products of the bath also coexist, such as: sulphuric acid, zinc sulphate, sulphate of magnesia, and so forth, organic materials and so on.

It results that according to the composition of the bath to be cooled, the crystallization of the sodium sulphate will start at a more or less high temperature and that, in order to obtain sufficient separation, it will be required to cool the bath treated at a more or less low temperature; it has been found that in practice it is always necessary to cool to at least 10° C., often even in the vicinity of 0° C., and sometimes below 0° C. The obtaining of such temperatures requires a source of cold of sufficiently low temperature in which it is sufficient to obtain a temperature of 10° C., it is rare that it is possible continually to have available the water at a temperature of from 0° to 5° C., for the cooling of the bath to 10° C.

Moreover it should be noted that in certain cases since in general, in factories a certain quantity of water has to be evaporated, that the bath treated to eliminate the sodium sulphate could be a bath previously concentrated.

Up to now the crystallization installations worked in conformity with two processes recapitulated hereinafter: First process: cooling of the bath by means of a cold-producing brine.

Then the factory has available a refrigerating machine cooling a cold-producing brine to the desired temperature and the brine circulates in the crystallization apparatus of known type.

It will be understood that with a view to reducing the consumption of units of cold, it is

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possible to cause the brine refrigerator to be preceded by another cooler wherein the cold is produced by the water or by the bath already cooled.

In spite of all, this process has the disadvantage of requiring a considerable amount of units of cold the cost of which is high. Moreover the temperature differences employed are relatively slight so that the surfaces of exchange must be considerable and since it is necessary for them to be of metal incapable of being attacked by sulphuric acid, the initial cost of the same and the cost of upkeep, is high. Second process: cooling of the bath by expansion and under very high vacuum.

If the bath to be cooled be brought into a space which is under very high vacuum, corresponding to an absolute pressure of a few millimeters of mercury, the bath evaporates owing to its sensitive heat and the heat liberated by the crystallization of Na_2SO_4, $10H_2O$. However, it will be understood that in order to ensure for example that the boiling temperature of the bath may be 10° C., considering that the boiling point of the bath is approximately 5° C., greater than that of water, the absolute pressure of the cooling apparatus must correspond to a boiling temperature of the water of 5° C. approximately that is, to 5 mm. of mercury.

The production of such a high vacuum can only be carried into effect by the well-known combination of a steam ejector combined with a vacuum pump according to an arrangement analogous to that diagrammatically illustrated in Fig. 1. The bath to be cooled arrives at 1 in a crystallizer exaporator 2 from where at 3 the cooled bath and the salt are extracted; this evaporator is connected at 4 with an ejector 5 fed with steam at 6, itself connected to a vacuum pump 7 through a condenser 8; 9 is a coldwater inlet and 10 a water return.

This apparatus has the great disadvantage of consuming enormous quantities of steam at the ejector considering the great volume occupied by the bath evaporation pump under such a low pressure.

With a view to reducing this steam consumption and without changing the principle hereinbefore referred to, the total expansion of the bath has been divided into several stages under increasing vacuum by providing at each stage a steam ejector appertaining thereto. In spite of all, the consumption of driving steam remains on the whole very high.

The present invention relates to a process in

which the cooling and the subsequent crystallization of the bath are obtained by placing as previously, the bath in a space wherein there is found a very high vacuum. But in this process the obtaining of the vacuum is entirely different.

The principle is known, of the sulphuric acid refrigerating machine wherein the cold is obtained by evaporation of any desired solution under a very high vacuum (the vapour emitted being absorbed by the sulphuric acid which is diluted owing to this, and which is concentrated later to be reintroduced into the circuit).

This process has not been often applied owing to the cost and the difficulties of reconcentration of the diluted sulphuric acid. The applicants have had the idea of applying this principle to the treatment of spinning baths with most advantageous results. For it is found that in artificial textile mills large quantities of sulfuric acid are employed, and this acid may be used, before being employed in the manufacture, for the absorption of the vapours emitted in the bath crystallization apparatus.

Fig. 2 shows a plan of apparatus which may be used for the application of the present invention. The bath to be cooled reaches at **11** a crystallization apparatus **12** by expansion under high vacuum of type known in itself and **13** designates the extraction of the bath and of the salt; this crystallizer **12** is connected through the conduit **14** to a sulphuric acid washer **15** wherein circulates the sulphuric acid fed at **16** and emerging at **17**. In this washer, the vapour produced by the evaporation of the bath owing to the heat yielded up by its own heat and by the heat from the crystallization of the sodium sulphate, is absorbed by the sulphuric acid. It results that the ejector **18** which produces the vacuum only has to carry along the gases incapable of condensation and not, as in the preceding process, all the evaporation vapour. Consequently, the consumption of steam of this ejector arriving at **19** will be infinitely small as compared with the consumption in the former process.

But this process presents another surprising interest. By way of example, it has been found that to obtain a temperature of 10° C., for the cooled bath, it would be sufficient to have an absolute pressure of 5 to 6 mm. of mercury; it will be necessary, it will be appreciated, that the pressure of the sulphuric acid circulating in **15** remains constantly lower than this value, that is for instance 4 mm. of mercury; according to the concentration of this acid, the latter may therefore have a temperature which would be for an acid of 70% H_2SO_4, 42°; 72% H_2SO_4, 47°; 74% H_2SO_4, 51°; 76% H_2SO_4, 55°; 78% H_2SO_4, 65°; 80% H_2SO_4, 70°; 82% H_2SO_4, 81°.

The calories liberated by the bath at a temperature of 10° will then be found in the sulphuric acid circulating in the absorber **15** at a much higher temperature and being able to be for example from 70° to 80° C. with acid at 80–82% H_2SO_4.

Therefore at the same time revalorization has been affected of the liberated calories which were unusable when they were available at a temperature of 10° C., but which may be used very readily at a temperature of 70–80°. They may be used for any desired purpose, for example by absorbing them by a bath circulation, or by a water circulation, in such manner as to obtain hot water for which there is always a call in such factories. In the figure, **21** designates a circulation pump for the sulphuric acid, **22** is an extraction of the diluted acid, **23** a feed of fresh acid and **24** a circulation of liquid to be heated in the exchanger **20**.

Thus by this process not only the steam consumption is reduced to a great extent, but also revalorization is effected of the calories liberated by the low-temperature bath.

The preceding description has been given by way of example: it is understood that this invention remains valid if the absolute pressure be different from that taken as example.

The expansion apparatus could likewise be divided into several stages working under increased vacuum without thereby altering the principle. The bath could likewise be partially cooled prior to its final cooling in the installation hereinbefore described.

The ejector, that is to say, the device extracting the gases incapable of condensation, could be replaced by an apparatus especially for this purpose; a vacuum pump of any suitable type.

I claim:

1. A process for the elimination of sodium sulphate from viscose coagulation baths, comprising passing the bath containing the sodium sulphate through at least one reduced pressure zone for effecting evaporation and cooling thereof, effecting an absorption of the heat of the vapors due to evaporation by sulphuric acid, maintaining a reduced pressure in respect to the sulphuric acid, and regaining the heat absorbed by the sulphuric acid for utilization thereof.

2. A process according to claim 1, wherein the sulphuric acid has a relatively high temperature depending on its concentration for the purpose of facilitating the recovery of the absorbed vapor heat.

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