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PROCESS OF PRODUCING WATERPROOF FIBROUS SHEETS

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6 Claims. (Cl. 92—21)

This invention relates to a process for the production of water-tight and waterproof papers and pasteboards by addition to the pulp or cellulosic material in the hollander or in the mixing vat of water-repellent or impregnating medium such as asphalt in the form of a very fine aqueous dispersion or emulsion produced or stabilised with the aid of caseinate, with or without the addition of resin size or similar sizing material, followed by precipitation with alum or other precipitant.

According to the invention there is added to the caseinate, to the finished dispersion or emulsion, or to the pulp, before precipitation, a slurry of calcium hydroxide or hydroxide of other earth alkali.

This addition of hydroxide does not in itself effect precipitation of the emulsion, but it promotes the precipitation by means of alum.

In practice, we first produce a very fine aqueous dispersion of asphalt as by the method described in the specification of United States Letters Patent No. 1,643,675 and we mix this dispersion by stirring, while in still warm condition, with the quantity of alkali caseinate necessary for stabilisation, i. e., between 2 and 5% reckoned on the quantity of the aqueous dispersion. Thereby there is obtained not only the required stability but also fining of the particles. It is, however, also possible to introduce the caseinate in the production of the dispersion, in suitable quantity, along with a selected emulsifier. Both ways lead to the necessary fine dispersion.

It is also advantageous if to the aqueous dispersion prepared in this way there is added resin size usual in sizing paper in quantities of about 2 to 4% reckoned on the paper material. But this is not necessary for success.

Such a fine aqueous dispersion if added alone to the paper stock would in the treatment of the paper stock run off in large quantities with the back-water, so that considerable losses of impregnating medium would result.

In order to prevent this, according to the invention there is added to the pulp or paper material in the hollander or to the caseinate or to the finished dispersion, but preferably to the pulp or paper material, milk of lime (calcium hydroxide) in such quantity that the content of lime reckoned as $Ca(HO)_2$ amounts to 0.15 to 0.85% reckoned on the paper material. The quantity of lime added depends on the hardness of the water, the nature of the paper material, the degree of grinding the same, and the quality of the dispersion used. After the addition of the lime has been effected and after good mixing, there is added a quantity of dispersion which corresponds to the contemplated content of impregnating medium in the finished product, and good mixing is again effected. At this time there are no changes of any kind in the mixture of substances.

Now a solution of alum is added to the mixture of substances in such quantity that an acid reaction occurs; blue litmus paper should become distinctly red. On the addition of the alum solution the material appears floccúlent and compact, the particles of impregnating medium, which before the addition of the alum run off with the water on pressing the material, adhere between and to the paper material, and the water runs off clear. Nevertheless, as may be determined microscopically, the particles of impregnating medium do not become coarser. Apparently, the colloidal precipitate of calcium sulphate and aluminium hydroxide produced on the addition of alum and distributed between and on the fibres of the paper material also envelops and holds fast the fine particles of impregnating medium.

Paper material prepared in this way when worked on the paper machine allows only very few bitumen particles to pass through the wire, so that the losses thereof are reduced to a minimum and the back-water is almost clear.

The small quantities of fibres and bitumen particles settle very rapidly, so that the back-water clears quickly and the material intercepted can be readily removed from the filters.

The material so prepared can be worked readily and furnishes, after melting of the impregnating medium at the dry end of the machine, a closed uniform product which, depending on the quantity of impregnating medium used, is water repellent or watertight and waterproof. It is surprising in this connection that bituminised papers so produced are also airtight, this being a property which otherwise is only obtainable with difficulty. This is doubtless attributable to the action of colloidal precipitation occurring on the addition of alum in which also the caseinate in the aqueous dispersion participates.

The technical advantages of this process reside in the possibility of employing very fine aqueous dispersions of the impregnating medium, in its marked retention in the paper material, notwithstanding its great fineness, in its uniform distribution, in the improvement of the finished product thereby effected, and in the obtainment of back-water which contain only small quanti-

ties of bituminous materials and fibres and which clear readily and rapidly.

We claim:

1. A process which comprises mixing a fibrous pulp material and an aqueous bituminous emulsion in which the emulsifying agent consists of an alkali hydroxide, said emulsion being stablized with an alkali caseinate, mixing said pulp material with at least approximately 0.15 per cent of calcium hydroxide reckoned on the paper material, the amount of calcium hydroxide being insufficient to precipitate said stabilized emulsion and adding alum whereby said emulsion is precipitated and the reaction products of the alkali caseinate, calcium hydroxide, and alum envelop the precipitated particles of bitumen and cooperate to produce a waterproof felted fibrous product.

2. In a process for the production of a felted fibrous material which comprises, mixing a casein stabilized bituminous emulsion with a paper pulp, adding alum to precipitate the bitumen of said emulsion on the fibers of said pulp, and forming a felted fibrous mat from said pulp material, the step of promoting said precipitation with said alum by incorporating an earth alkali hydroxide in said paper material prior to said precipitation in an amount sufficient to promote but insufficient to effect said precipitation, whereby upon precipitation of the bitumen a colloidal precipitate of earth alkali sulfate and aluminum hydroxide envelops the precipitated particles of bitumen.

3. In a process for the production of a felted fibrous material which comprises, mixing a casein stabilized bituminous emulsion with a paper pulp, adding alum to precipitate the bitumen of said emulsion on the fibers of said pulp, and forming a felted fibrous mat from said pulp material, the step of promoting said precipitation with said alum by incorporating calcium hydroxide in said paper material prior to said precipitation in an amount sufficient to promote but insufficient to effect said precipitation, whereby upon precipitation of the bitumen a colloidal precipitate of calcium sulfate and aluminum hydroxide envelops the precipitated particles of bitumen.

4. In a process for the production of a felted fibrous material which comprises, mixing a casein stabilized bituminous emulsion with a paper pulp, adding alum to precipitate the bitumen of said emulsion on the fibers of said pulp, and forming a felted fibrous mat from said pulp material, the step of promoting said precipitation with said alum by incorporating not less than approximately 0.15 per cent, reckoned on the paper material, of an earth alkali hydroxide in said paper material prior to said precipitation, the amount of said earth alkali hydroxide being insufficient to effect precipitation of said emulsion in the absence of said alum, whereby upon precipitation of the bitumen a colloidal precipitate of earth alkali sulfate and aluminum hydroxide envelops the precipitated particles of bitumen.

5. In a process for the production of a felted fibrous material which comprises, mixing a casein stabilized bituminous emulsion with a paper pulp, adding alum to precipitate the bitumen of said emulsion on the fibers of said pulp, and forming a felted fibrous mat from said pulp material, the step of promoting said precipitation with said alum by incorporating not less than approximately 0.15 per cent, reckoned on the paper material, of calcium hydroxide in said paper material prior to said precipitation, the amount of said calcium hydroxide being insufficient to effect precipitation of said emulsion in the absence of said alum, whereby upon precipitation of the bitumen a colloidal precipitate of calcium sulfate and aluminum hydroxide envelops the precipitated particles of bitumen.

6. A process for the production of a waterproof felted fibrous paper material which comprises, mixing a casein stabilized aqueous bituminous emulsion with a paper pulp, precipitating the emulsion on the pulp fibers with alum, and forming a felted fibrous mat, said precipitation being effected in the presence of from approximately 0.15 to approximately 0.85 per cent of calcium hydroxide reckoned on the paper material, whereby said calcium hydroxide promotes the precipitating action of said alum and a colloidal precipitate of calcium sulfate and aluminum hydroxide envelops the precipitated particles of bitumen.

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