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# ALIEN PROPERTY CUSTODIAN

## PROCESS FOR THE PREVENTION OF RESINOUS PRECIPITATIONS IN PAPER-MAKING

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This invention relates to a process for the prevention of resinous precipitations from the cellulose used in paper-making.

The greater part of cellulose serving as initial material in paper-making contains according to the employed raw material and the process of regeneration varying amounts of resin. Especially the cellulose obtained by the sulphite process or from fresh wood contains sometimes more than 1% of resin proving very disadvantageous in the following manufacturing process, particularly of fine paper sorts, since the resin precipitates as a sticky substance in the edge mill, crusher, called "Wurster", rag-engine and at the paper sieve of the engines, leading to various difficulties and interruptions in the manufacturing process.

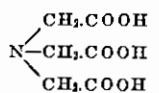
It has already been endeavoured to eliminate these difficulties by adding to the cellulose adsorbing agents, such as kaolin, which should reduce the stickiness of the resinous substances. The presence of kaolin, however, is undesired in many paper sorts and the precipitation of resin itself cannot be prevented by such additions.

Acid-eliminating agents have also been proposed for the present purposes. By their addition the stickiness of the resinous substances is, however, still increased. For this reason endeavours have also been made to add to the paper pulp alkaline reacting or alkali-eliminating agents which by saponifying the resins balance the disadvantageous action of the latter ones, but on the other hand effect—because used in excess—undesired swelling of the cellulose. Besides, the free alkali must again be neutralized by increased addition of an acid or of aluminium sulphate, which leads to high precipitations of resin-acid or resin soap. Finally the foaming of the dissolving or emulsifying resinous compounds is a disagreeable phenomenon.

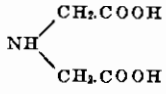
Now it has been found that the precipitations of resins and resinous substances, such as hemicelluloses, xylianes and the like, can be prevented by adding to the cellulose aqueous solutions of the salts of phosphoric acids in amounts of generally not more than 1% of the cellulose weight, whereby neither the usual manufacturing process is affected in any way, nor the paper fibre or the pH-value considerably influenced. For the effectiveness of the present process it is above all important not to exceed the maximum amount of about 1% in the case of alkaline reacting salts and about 2-3% in the case of acid salts, because upon additions beyond the mentioned percentages the emulsifying and saponifying action which is characteristic for the phosphates begins, eliminating the saponifiable substances, such as the resin, out of the substrate, thus making them removable by washing. In the present process such a phenomenon is, however, undesired, be-

cause it affects the swelling of the paper fibre as well as the pH-value and causes the foaming of the mass, whereas the disagreeable resin precipitations do not cease. Upon additions of less than 1%, especially 0.1 to 0.4%, of the dry weight of cellulose, resin and resinous substances are no longer precipitated from the fibre, but remain in homogeneous distribution on it.

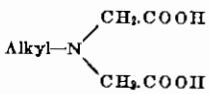
Besides the salts of phosphoric acids, the water-soluble salts of aminocarboxylic acids, containing more than one carboxylic acid group per amino nitrogen atom, standing in  $\alpha$ -position, thereto, and containing also an organic radical, but free from an alkyl or cycloalkyl radical having 10 or more carbon atoms when the whole molecule contains only 2 carboxylic acid groups, have proved effective in the process according to the present invention. As representatives of the simplest compounds which are obtainable by acting with ammonia on monochloroacetic acid or by saponifying nitriles be mentioned the trimethylamine- $\alpha$ -,  $\alpha'$ -,  $\alpha''$ -tricarboxylic acid of the formula:



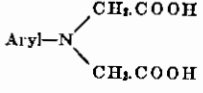
the iminodiacetic acid of the formula:



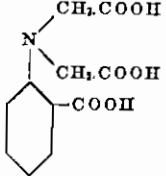
and such iminodiacetic acids, in which the hydrogen atom of the imino group is substituted by an alkyl, aryl, aralkyl or oxyalkyl group, such as the alkyliminodiacetic acid of the formula:



or the aryliminodiacetic acid of the formula:



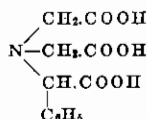
Further the anthranilic-N,N-diacetic acid of the formula:



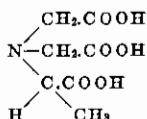
which contains in the aryl nucleus an acid group standing in ortho-position to the carbon atom to which the imino group is attached.

By replacing the hydrogen atoms of the methylene group of the above trimethylamine- $\alpha$ -,  $\alpha'$ -,

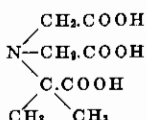
$\alpha''$ -tricarboxylic acid by alkyl or aryl groups suitable compounds are obtained, such as C-phenyl-trimethylamine- $\alpha$ -,  $\alpha'$ -,  $\alpha''$ -tricarboxylic acid of the formula:



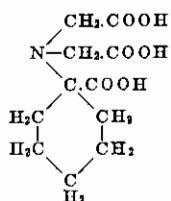
C-monomethyl-trimethylamine- $\alpha$ -,  $\alpha'$ -,  $\alpha''$ -tricarboxylic acid of the formula:



C-dimethyl-trimethylamine- $\alpha$ -,  $\alpha'$ -,  $\alpha''$ -tricarboxylic acid of the formula:



N-(1-carboxy-cyclohexyl)imino-diacetic acid of the formula:



The water-soluble salts of phosphoric acids can be applied in any degree of hydration. The preferred alkali metal and ammonium salts are those with a favourable pH-value, such as mixtures of mono- and disodium orthophosphate or of disodium orthophosphate and tetrasodium pyrophosphate. Mixtures containing sodium hexametaphosphate are also suitable for the present purposes. As especially effective have proved the water-soluble salts of polyphosphoric acids, above all of tripolyphosphoric acids,

which have not only a favourable pH-value for the regeneration, but above all react advantageously in presence of hard water as well as upon the additions of aluminium salts and resin-sizing usual in paper-making. The phosphate additions can also be combined with acids, alkalis and adsorbing agents, such as kaolin, bentonite, water-glass and talc, which do not influence the intended effect.

*Example 1.*—250 kg of cellulose with more than 1% of resin are treated 1 hour in the edge mill with 1 kg of sodium tripolyphosphate dissolved in 10 litres of water and with about double the quantity of water from the dry weight of cellulose and then worked in the usual manner in the rag-engine, mixing through and paper engine which all remain free from resin. Old resin deposits on walls, stones and knives are gradually dissolved and can be removed without difficulty from the sieve. Engine parts with hard resin deposits are brushed before with a hot solution of 10 g per litre of sodium tripolyphosphate  $\text{Na}_5\text{P}_3\text{O}_{10}$ .

*Example 2.*—The cellulose passed through the edge mill or the crusher is prepared on the rag-engine with water in the proportion of 1:20. 1 kg of sodium tripolyphosphate  $\text{Na}_5\text{P}_3\text{O}_{10}$ , dissolved in 10 litres of water, is slowly added.

The phosphate additions can be made in the rag-engine or in any of the following manufacturing states, the effect obtained being the same as upon addition in the edge mill.

*Example 3.*—250 kg of cellulose are prepared with 0,5 kg of sodium tripolyphosphate  $\text{Na}_5\text{P}_3\text{O}_{10}$ , 2,5 kg of talc and double the quantity of water from the dry weight of the cellulose and passed through the edge mill as usual.

*Example 4.*—To 100 kg of cellulose with about 1,2% of resin, prepared with about double the quantity of water, are added 0,4 kg of the sodium salt of trimethylamino- $\alpha$ -,  $\alpha'$ -,  $\alpha''$ -tricarboxylic acid. After a treatment of about 1 hour the mass is drawn off and then worked in the usual manner in the edge mill, rag-engine, mixing through and paper engine.

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