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ALIEN PROPERTY CUSTODIAN

PROCESS FOR THE PREPARATION OF DYESTUFFS OF THE ANTHRACENIC SERIES

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It is known that the condensation of 9.10 disulphuric esters of leuco-anthraquinones containing an amino group acylated or not, in the beta position, or of the salts of the said esters, can be carried out in an alkaline medium by means of various oxidizing products or salts which have the property of acting as such when passing at the lower valency. The products of the condensation are esters of the N-dihydro-1.2.2'.1' anthraquinone.azine or of its substituted derivatives, which have a great value from the tinctorial point of view, especially in printing.

All oxidizing products up to now proposed have more or less evident disadvantages. Some have an action that is not limited to condensation only, others produce salts which are partially soluble in an alkaline medium, when they have given off one atom of oxygen. These salts contaminate the dyestuff, which necessitates an ulterior purification.

The present invention has for its object the use of silver oxide as agent for the condensation of the leuco-esters of the 2-amino-anthraquinone and of its substituted products (position 1 remaining free). The oxidizing products used previously, were known condensation agents, such as potassium permanganate, ferric chloride, sodium hypochlorite, potassium ferricyanide, potassium persulphate or lead peroxide. On the contrary, silver oxide has never been used up to now as a condensation agent. Now, according to the invention, it has been found that silver oxide permits not only to condense two molecules of di-ester with a sufficient yield, but also to completely recover the silver, the product of the decomposition of the oxide being completely insoluble in alkaline medium, after reaction.

The oxygen evolved during the reaction does not act upon the organic molecule, but combines itself with the two atoms of hydrogen freed by that one, to form water. The beta-amino anthraquinone sulphuric di-ester, or its substituted derivatives, are soft reducing agents of the silver oxide and metallic silver at a granulated state is obtained at the end of the reaction. A silver mirror is not formed at any moment, the grey powder adheres slightly to the walls of the vessel in which the condensation takes place and even gets loose at the end of the reaction.

The condensation can be accomplished either with dry silver oxide, or recently prepared and as a paste, either with its ammoniacal solution, or also with silver oxide formed directly in the mixture which will enter into reaction. In this case, it is possible to start indifferently from soluble or not soluble silver salts, able to give oxide in an alkaline medium.

The totality of the silver can be recovered after the reaction, and can easily be transformed into a soluble salt, which permits an easy regeneration of the oxide and the elimination of small proportions of organic products.

The following are non limiting examples for the application of the invention.

Example 1

500 parts of water, 20 parts of sodium carbonate and 18 parts of beta-amino-anthraquinone sulphuric di-ester are heated at a temperature of 100°C, and 14 parts of silver oxide are then charged in small equal portions. The temperature is maintained till the end of the transformation, the mixture is cooled, filtered and the silver precipitate is washed. The dyestuff, as tetra-ester, is in solution in the filtrate. It is isolated in the usual manner.

Example 2

450 parts of water, 50 parts of caustic soda of 30% and 18 parts of 2.amino.3.chlor.-anthraquinone sulphuric di-ester, are heated at a temperature above 80°C and 10 parts of silver oxide are gradually charged (as a paste). The temperature of the mixture is maintained a few minutes and the process is then carried out as in Example 1.

Example 3

A solution of 30% of silver nitrate (65 parts) is slowly and regularly poured in a hot solution composed of 450 parts of water, 65 parts of caustic soda of 30% and 18 parts of 2.amino.3.chlor.-anthraquinone sulphuric di-ester. The temperature of the mixture is maintained till the end of the reaction. The mixture is filtered after cooling and the dyestuff is isolated in the usual manner.

The silver precipitate is then treated by diluted nitric acid, in the hot. The solution, filtered in order to eliminate the organic products, can be concentrated in order to recover the salt in a solid state or neutralized and used directly for a new condensation.

Example 4

14 parts of silver chloride, as a paste, are charged gradually in small equal portions, in 400 parts of water, 40 parts of caustic soda of 30% and 14 parts of 2.amino.3.chlor.-anthraquinone

sulphuric di-ester, heated above 80°C. The temperature is maintained a few minutes and the dyestuff, after cooling and elimination of the silver, is isolated.

Example 5

480 parts of water, 60 parts of caustic soda of 30%, 7 parts of 2.amino.3.chlor.-anthraquinone sulphuric di-ester, are heated under pressure at 120°C during 15 minutes. After elimination of the silver, and precipitation by a mineral acid, the dyestuff gives a blue shade in the vat dyeing.

Example 6

In a solution, maintained at a temperature above 80°C, which contains 400 parts of water, 32 parts of caustic soda of 30% and 7 parts of 2.amino.3.chlor.anthraquinone sulphuric di-ester, is poured a solution prepared with 100 parts of water, 17 parts of caustic soda of 30%, 10 parts of silver nitrate and 20 parts of ammonia of 25%. The isolation of the dyestuff is carried out as in the preceding examples.

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