

ALIEN PROPERTY CUSTODIAN

MANUFACTURE OF MATERIALS COMPRISING ARTIFICIAL RESINS

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For various purposes the problem exists in the art to work up artificial resins and similar bodies in a dispersed form. Hitherto in principle the path followed has always been to disperse more or less initially condensed resins, that is to say, to dissolve, to emulsify or to suspend the same.

According to the present invention on the other hand the substances causing the resin formation are dispersed individually and the condensation first carried out in the additive system, that is to say, in the simultaneous presence of other indifferent substances taking no part in the resin formation, upon or in which the condensation or polymerization product is intended to be dispersed, without in this manner the yield being reduced to any appreciable extent from a commercial point of view by evaporation losses of one of the resin forming components.

Proposals have already been made to carry out the condensation by introduction of the individual reaction components for example into a mass of fibres, (compare German Patent No. 231,148). According to this latent phenol and formaldehyde are employed. In this manner there occur, however, unavoidable losses of formaldehyde unless special devices are provided for the recovery of the formaldehyde evaporation losses.

Such evaporation losses are avoided according to the present new process in such a manner that according to the invention advantageously aqueous dispersions of phenol and of components producing artificial resin formation therewith, which possess a higher evaporation point than the dispersing agent, for example compounds splitting off aldehyde such as hexamethylenetetramine, are brought together in the presence of the indifferent body or body to be coated and, for example by heating, condensed and polymerized in the open atmosphere, that is to say, without recovery devices for the artificial resin components, during which suitably at the same time the dispersing agent is removed, for example gradually evaporated.

Instead of the phenols obviously also their homologues or other substances giving resin formations with aldehyde or their substituents can be employed, as for example cresols, primary amines, as for example aniline, urea, casein, glutin-containing substances such as gelatine, glue etc. Instead of the compounds splitting off aldehyde also such substituents can be employed as produce resin formation, with phenol or its substituents and the evaporation point of which is so high that at the temperature necessary for the condensation no appreciable evaporation losses

take place, as, for example, sulphur which with phenol gives the known phenol-sulphur resins. The compounds splitting off aldehyde can be employed in the form of their isolated previously formed compounds, as for example in the form of the previously mentioned hexamethylenetetramine, or also by combination of equivalent quantities of aldehyde and ammonia, which thereby intermediately come into application in the course of the complete working process.

It has already been known for a long time to harden phenol-aldehyde condensation products or other products condensed with phenol by additions of hexamethylenetetramine. Recently it has also been proposed for the impregnation with artificial resins of large porous bodies such as balks of timber etc. to carry out the condensation of phenol from the beginning with formaldehyde-ammonia (compare Swiss patent 141,109).

Furthermore, it has also been proposed to carry out the condensation of phenol with aldehyde-ammonia with the addition of solvents, in fact either with such solvents in which the condensation product is intermediately dissolved, that is to say organic solvents (compare British Patent 6,363/1912), or of water in such quantities that the aldehyde-ammonia and the phenol for homogeneous admixture are initially dissolved therein (compare U. S. Patent 1,187,230/1916).

None of the patents mentioned has however anything to do with the process of the present invention and they are distinguished from the latter by the two following essential features:

1. In the previous processes the condensation is carried out as an independent separate working operation.

2. The previous processes required the application of reflux devices for the evaporating solvent. According to the mentioned patents thus in the first place a non-dispersed starting material is obtained, which then in a second stage is subjected to a further working up (as coating or impregnating material in dissolved form for the manufacture of cast or pressed objects, if desired with the subsequent addition of filling materials etc.).

In contradistinction to these prior processes according to the present invention the result is attained that no essential losses arise by evaporation of the components taking part in the resin formation. In addition it is now also possible to carry out the condensation in excellent yield in any suitable dilution and distribution also in the open atmosphere, that is to say without any devices for recovery of constituents which distil off.

This result is attained on the one hand in such a manner that, as already stated, as components for artificial resin formation for example compounds splitting off aldehyde, such as formaldehyde-ammonia, hexamethylenetetramine, sulphur and the like substances are employed, which possess a higher evaporation point than the dispersing agent and thus for example at the point of evaporation of water possess a very low vapour pressure and also for example are not volatile in steam, and is attained on the other hand in such a manner that the reaction velocity of these substances with phenols and the like even at great dilutions is still sufficiently large in order to bind the phenols so rapidly that no essential losses of the latter substances by evaporation can any longer take place.

By this means therefore the hitherto customary initial condensation as an independent working operation is obviated and the condensation is only carried out and localised at the place where the finished resin is finally intended to be employed. By this means also the result is attained that the resin produced is obtained immediately and without special operations distributed in a dispersed form on or in the indifferent bodies. This is particularly valuable in such cases in which is concerned the carrying out of the condensation on a body in a thin layer, as for example in a paper or fabric web saturated with the dispersion.

In particular instances there accrues in addition the very important advantage that particular dispersions of this type, as for example formaldehyde-ammonia, can be employed in completely neutral solutions and therefore can be associated with the most varied substances, even alkali-sensitive bodies as for example wood fibres, straw fibres, cotton etc. In this case there always exists a neutral or only very weakly alkaline reaction of the system since the alkaline ammonia, which gradually becomes free as the reaction proceeds, vaporises at the temperatures necessary for the reaction to the same extent as it is produced.

By dispersion is to be understood any type of distribution, that is to say solution, emulsion or fine or coarse suspension. As dispersing agent advantageously water is employed. In this case the components taking part in the resin formation can either at the same time be distributed and employed in one and the same dispersing agent, or a dispersion of each individual component in a dispersing agent is produced, for example a suspension of phenol and a solution of hexamethylenetetramine. In this case also at the same time different liquids as for example water and alcohol can be employed. These preparations of the components can be added separately, for example pressed or sucked through the fibrous material serving as a felting skeleton or filling material, or they can be combined with loose fibrous material.

The condensation of the resin components can suitably also be caused to take place and assisted by additional treatment with chemicals as for example alkalies, catalysts and the like.

The removal at least of a part of the dispersing agent contained in the system can be carried out by pressing off, sucking off, centrifuging, cataphoresis and the like.

The surface on which the resin is intended to be condensed and retained can also be the interior of porous bodies as for example of wooden articles to be impregnated or perforated fibrous bodies such as paper or fabric webs, or also the

surface of individual loose fibres which are mixed with the dispersion of the binding agent components directly in the loose condition and subsequently, according to the fibres employed, simultaneously felted, freed from the liquid and hardened.

With quite particular advantage the working method of the invention can finally be employed in conjunction with the process of ——— to which masses similar to wood are produced in such a manner that feltable fibrous substances are felted and before, during or after the felting treated with preparations of binding agents capable of being hardened later, whereupon the excess liquid is removed and the fibrous mass suitably at the same time shaped in a manner which does not entirely remove the porosity and, likewise with retention of the porosity, dried at ordinary or elevated temperature with hardening of the binding agent.

The following examples illustrate the invention.

Example 1

To 900 parts of water are added about 50 parts of about 40% formalin solution and also about 30 parts of 25% ammonia. This solution is then suitably neutralised for example with a little aluminium sulphate or the like. Into this solution are introduced 10 parts of dry mechanical wood pulp or cellulose. This material is well suspended and finally after the addition of about 5 parts of cresol the whole stirred until the cresol is completely distributed in the mass. Then the mixture is formed into webs for example on a paper machine. The remaining solution is then together with the paper web heated to about 80–100° C. and slowly dried. In this condition a condensation has already taken place. Subsequently any suitable further treatment can take place, for example the final hardening produced in hot presses.

Example 2

Dry paper sheets or fabric webs are immersed in a solution of 30 parts of hexamethylenetetramine, 100 parts of phenol and 900 parts of water. The saturated sheets or webs are then heated to 80–90° C. and slowly dried. For hardening of the resin they are then individually, or pressed several on top of one another, maintained for some time at a temperature of 120–140° C.

Example 3

In the same solution as in Example 2 are immersed well dried and if desired evacuated wooden objects, for example planks, balks, etc. until the solution has penetrated well. These wooden objects are then likewise first heated to 80–90° C. slowly dried and hardened at 120–140° C. The penetration of the impregnation solution into the wood can in this case likewise be facilitated by a slight excess of ammonia.

Example 4

Porous shaped bodies of asbestos fibres, slag wool or the like are immersed in the following liquid; 100 parts of crude cresol dissolved in 900 parts of water with the addition of the necessary quantity of caustic soda lye, contain 40–50 parts of finely divided sulphur. The saturated shaped bodies are heated to 80–90° C. and slowly dried, thereupon the heating is continued at 120–140° C., if desired with repeated drying and pressing until the hardening is sufficient.