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ROSIN SIZED PAPER PREPARED FROM CYANOALKYLATED WOOD PULP AND METHOD OF PRODUCING SAME

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This invention relates to the preparation of paper, and particularly to the preparation of paper of unusual sizing properties, from cellulose materials.

It is a primary object of the invention to prepare exceptionally hard sized paper from cellulosic materials with the use of conventional sizing agents.

Still further objects and advantages of the invention will appear from the following description and appended claims.

These and other objects of the invention can be obtained by employing as the raw material for the manufacture of the paper cellulosic material which has been modified by mixing or reacting it with an agent capable of adding cyanoalkyl groups. This can be accomplished most suitably by mixing ordinary wood pulp in the form of an aqueous pulp suspension with suitable quantities of acrylonitrile or methacrylonitrile, to which preferably has been added a small amount of a polymerization inhibitor, such as hydroquinone, ethyl or methyl ether of hydroquinone, tertiary butyl catechol, or the like, in the presence of an alkaline catalyst, such as one of the alkali metal hydroxides, including preferably caustic soda. The reaction may in general be carried out in the following manner.

A typical pulp suspension is prepared by stirring one part by weight of dry pulp in at least twenty-five parts by weight of water for a period of 1 hour or more, while maintaining the mixture at 20 to 65° C. After adding from one and one-half to three parts by weight of 50% caustic soda or other alkali metal hydroxide, the suspension is held for 10 to 60 minutes at a temperature between 20 and 65° C., during which time the pulp swells. The pulp is then filtered or centrifuged, after which it is placed in a suitable vessel and from 20 to 40 parts by weight of acrylonitrile or methacrylonitrile are added. The mixture is held at 40 to 80° C. for 1 to 2 hours, after which it is filtered and washed to free the pulp of acrylonitrile and any caustic soda remaining, and finally dried. The pulp thus obtained usually contains between 4 and 7% nitrogen.

When a dilute aqueous slurry is prepared from the dry pulp prepared as hereinbefore described and the slurry is treated in the usual manner with small quantities of conventional paper size and an aluminum salt, such as aluminum sulfate or aluminum chloride, hand sheets can be prepared from the pulp so treated which show as much as a tenfold increase in sizing, as measured by the standard ink penetration test, as compared with hand sheets prepared from the same pulp which has not been modified in the manner herein described.

A further understanding of the invention will be obtained from the following examples, parts and percentages being by weight unless otherwise specified.

Example I

Fifty parts of Gatineau bleached sulfite wood pulp were stirred into 2000 parts by volume of water in a suitable reaction vessel over a period of two hours, while

maintaining the mixture at 30 to 60° C. Eighty grams of 50% NaOH were added to the resulting slurry, after which it was held for 15 minutes at 60 to 65° C. The pulp was then filtered and returned to the reaction vessel, after which 1000 parts by volume of acrylonitrile inhibited with 1% hydroquinone were added. The mixture was held at 55 to 65° C. for one hour, after which it was filtered, and the resulting pulp was washed with cold and hot water until it was free from acrylonitrile and caustic soda, and then air dried. The resulting dry pulp was found on analysis to contain between 4 and 5% nitrogen.

The dry pulp obtained as described in the preceding paragraph was made into a 2% slurry with water after which 1% of partly neutralized rosin size which has been fortified with maleic anhydride, and 3% of aluminum sulfate were added on the weight of the dry pulp, and the resulting slurry made into hand sheets on a Noble and Wood sheet making machine. The resulting hand sheets were conditioned at 70° F. and 50% relative humidity for 24 hours, after which they were subjected to the standard ink penetration tests for measuring degree of sizing. On completion of the test the sheets showed 1000 seconds sizing.

When hand sheets were prepared in the same manner from a 2% slurry of Gatineau bleached sulfite wood pulp which had not been modified in the above-described manner were tested, the sheets showed only 100 seconds sizing, when tested by the ink penetration method.

According to the standard ink penetration method 2-inch squares are cut from the prepared hand sheets, and the squares are floated upon a bath of ink. The time which is required for the ink to uniformly penetrate the floating squares is then recorded, with longer times representing proportionate increases in sizing.

Example II

Fifty parts of unbleached kraft pulp were stirred into 2000 parts by volume of water in a suitable reaction vessel over a period of two hours, while maintaining the mixture at 30 to 60° C. One hundred grams of 50% NaOH were added to the resulting slurry, after which it was held for 15 minutes at 60 to 65° C. The pulp was then filtered and returned to the reaction vessel, after which 700 parts by volume of acrylonitrile inhibited with 0.1% methyl ether of hydroquinone were added. The mixture was held at 55 to 65° C. for one hour, after which it was filtered, and the resulting pulp was washed with cold and hot water until it was free from acrylonitrile and caustic soda, and then air dried. The resulting dry pulp contained between 2 and 3% nitrogen.

The dry pulp obtained as described in the preceding paragraph was made into a 2% slurry with water, after which ¼% of completely neutralized dry rosin size and 3% of aluminum chloride were added on the weight of the dry pulp, and the resulting slurry made into hand sheets on a Noble and Wood sheet making machine. The resulting hand sheets were conditioned at 70° F. and 50% relative humidity for 24 hours, and were found to be exceptionally hard sized and of improved printability as compared with paper prepared from unbleached kraft pulp which had not been modified in the manner described above.

A wide variety of pulps may be cyanoalkylated and used to make hard sized paper according to this invention, including unbleached sulfite pulp, bleached and unbleached kraft pulp, soda pulp, hardwood pulp, and mixtures of groundwood pulp with unbleached kraft pulp and other pulps. It is also possible to employ to advantage pulps containing as little as ½% nitrogen and as much as 10% nitrogen.

The sizing agents employed may be ordinary rosin size

dispersions of the type consisting of rosin dispersed in water with the aid of caustic soda, or fortified sizes may be used, in which the rosin is fortified with such materials as maleic acid or anhydride, fumaric acid, polystyrene, wax, vinyl acetatecrotonic acid copolymers or styrene-maleic acid or anhydride copolymer. According to the examples, it is possible to prepare highly sized paper with as little as ¼% dry size solids on the weight of the dry pulp. In some instances as much as 5% size may be used to advantage. It is also possible to add instead of 3% aluminum sulfate or chloride any amount between 1 and 7% on the weight of the dry pulp.

Any suitable paper-making apparatus can be used instead of the Noble and Wood machine, depending on the scale of operations desired. Other conventional variations in the sheet making procedure coming within the scope of the appended claims will occur to those skilled in the art.

What is claimed is:

1. The method of making paper which comprises first preparing an aqueous suspension of cyanoalkylated wood pulp, mixing with said suspension small amounts of a rosin size and an aluminum salt selected from the group consisting of aluminum sulfate and aluminum chloride, and then forming the suspended material into paper.

2. The method of preparing sized paper which comprises first preparing an aqueous suspension of cyanoalkylated wood pulp, adding an aqueous dispersion of a rosin size to the pulp in an amount sufficient to provide from ¼ to 5% of dry size based on the dry weight of the pulp, then admixing with the pulp a small amount of aluminum sulfate, and forming the pulp into paper.

3. The method of preparing sized paper which comprises first preparing an aqueous suspension of cyanoethylated wood pulp, adding an aqueous dispersion of rosin to the pulp in an amount sufficient to provide from ¼ to 5% of dry size based on the dry weight of the pulp, then admixing with the pulp from 1 to 7% of aluminum sulfate, and forming the pulp into paper.

4. Hard sized paper prepared from cyanoalkylated wood pulp and sized with a rosin size and an aluminum salt selected from the group consisting of aluminum sulfate and aluminum chloride.

5. Hard sized paper prepared from cyanoethylated wood pulp and sized with rosin and aluminum sulfate.

6. Hard sized paper prepared from cyanoethylated wood pulp and sized with rosin, fortified with maleic anhydride, and aluminum sulfate.

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