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SEALING OF DYED ANODIZED ALUMINUM

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The present invention relates to the sealing of dyed anodized aluminum.

Dyed anodic coatings on aluminum must be sealed in order to prevent staining, leaching of the dye in water, and possible loss of color by sublimation of the dye. In addition, it has been shown that certain sealing techniques enhance the lightfastness of the dyed coating.

Sealing can be accomplished by impregnating the film with oils and waxes; by hydrating the aluminum oxide, which causes an increase in volume thereby closing the pores; or by treatment with hydrolyzable metallic salts such as nickel acetate and cobalt acetate. The latter method is the one most commonly used for dyed coatings.

The hydrolyzable metallic salt sealing treatment or, as it is commonly referred to, the nickel acetate method, has the advantages of providing almost instantaneous setting of the dye, thereby minimizing bleeding, and of producing increased lightfastness with dyes which react to form a heavy metal complex. However, the hydrolyzable metallic salt sealing treatment is bound up with a serious disadvantage in that, when the film is completely sealed, a cloudy coating of basic nickel (or cobalt) acetate is deposited on its surface. In addition, on heavy anodic coatings dyed with concentrated dye solutions (such as for black work), a residue which appears to be a reaction product of the dye, the aluminum oxide, or impurities contained therein, and the hot water, is also formed. These films are referred to in the trade as "smut."

While the basic nickel acetate smut can be controlled to some degree by careful regulation of pH, sealing treatment time and temperature of the sealing bath, the dye smut left on black and other dark shades has heretofore been almost impossible to eliminate. The formation of these smuts necessitates an additional operation in the processing cycle, that of mechanically polishing the surface of the work (commonly referred to as "color buffing"). This is a costly operation for the anodizer, since each part requires individual handling and, in addition, there are numerous complicated shapes which are impossible to polish mechanically.

A primary object of the present invention is the embodiment of a process whereby sealing of dyed anodized aluminum may be accomplished by the hydrolyzable metallic salt sealing treatment but without the formation of smut, whereby the necessity for the aforesaid additional operation for the mechanical elimination of smut is obviated.

This object of the invention is realized by the expedient of incorporating into the sealing bath—which otherwise consists essentially conventionally of nickel acetate and/or cobalt acetate in aqueous solution also containing a pH determinant or buffer—a so-called "anti-smut" agent. As will hereinafter more specifically appear, the anti-smut agent takes the form of a lignosulfonate which may be used in a variety of forms, e.g. simply as straight sulfite waste liquor which has been evaporated to dryness, or in the form of a metal lignosulfonate such as calcium lignosulfonate or sodium lignosulfonate, or most advantageously in the form of desugared lignosulfonate, preferably desugared calcium lignosulfonate.

The process of the invention has the advantage of being quite uncomplicated. Thus, the aluminum article (and this includes articles of which the exposed surface or surfaces are of aluminum, the remainder of the articles being of desired composition) is anodized (anodically oxidized) and is thereafter dyed in a solution of an appropriate dyestuff, as in current commercial practice. The dyed article is then subjected to the desired sealing operation by immersion in an aqueous sealing bath which contains the nickel acetate and/or cobalt acetate and a buffer such as boric acid, borates, acetate, or other well known buffering agents. The proportions of these ingredients are widely variable and, generally speaking, this phase of the procedure may follow the teaching of Tosterud (U.S. Patent No. 2,008,733). The important feature, if the object of the invention is to be realized, is that the sealing bath also contains at least 0.1% by weight of anti-smut agent; the upper limit of content of this agent is determined solely by solubility characteristics and economics, any excess can do no harm except that its use may be wasteful.

In marketing the composition according to the invention, it is preferred to have the said composition in the form of a dry mixture, ready for incorporation into water to form a sealing bath. Such a dry composition may, for example, consist of substantially equal parts by weight of nickel acetate, buffering agent (e.g. boric acid), anti-smut agent (e.g. desugared calcium lignosulfonate), and about ⅕ part by weight of cobalt acetate. It is preferred to add this to water, when preparing a sealing bath, in a proportion of 16 grams per liter of aqueous bath. The buffer is used so that it determines a pH such that there is no precipitation in the bath of hydroxides of metals due to hydrolysis of the hydrolyzable nickel acetate and/or cobalt acetate. The necessary pH may be achieved by the addition of further boric acid to the sealing bath if necessary; it is also possible to establish the appropriate pH by the periodic addition of acetic acid to the sealing bath.

As already mentioned, the result here desired to be achieved involves the presence of the lignosulfonate molecule. This may be derived from a relatively pure salt, such as calcium lignosulfonate or sodium lignosulfonate or from a relatively impure material such as the product of the evaporation to dryness of ordinary sulfite waste liquor. The preferred material is desugared calcium lignosulfonate which presents the advantage over the undesugared lignosulfonates that, due to the minimum amount of sugar present, it is less subject to decomposition from growth of bacteria mold, and that a sealing bath made up from a mixture containing this desugared anti-smut agent requires no adjustment of pH when made up with normally neutral or slightly acid water.

Sulfite waste liquor which is available in large quantities in the manufacture of sulfite pulp comprises considerable quantities, in some cases up to about 20%, of sugar, and processes have been developed whereby these sugar values are effectively utilized for the manufacture of various ultimate products such, for example, as yeast (more especially to be used in the feeding of cattle) and ethyl alcohol. The residue from these processes—namely, desugared sulfite waste liquor, consisting essentially of calcium, magnesium, sodium and/or ammonium lignosulfonates, depending upon the base material used in making the cooking liquor—is thus available cheaply and in large quantities. The dry residue is obtainable by evaporation.

The following examples illustrate representative embodiments of the invention, it being understood that the embodiments employing the aforesaid desugared material

are the presently-preferred embodiments. In the said examples, the parts unless otherwise indicated are by weight, and the relationship of parts by weight to parts by volume is the same as that between grams and milliliters.

Example 1

A homogeneous dry mixture is made up by intimately admixing 2.5 parts of nickel acetate crystals, 0.5 part of cobalt acetate crystals, 2.5 parts of boric acid and 2.5 parts of dry desugared calcium lignosulfonate. The mixture is stable on storage for long periods of time.

In use, the anodically oxidized and dyed article is immersed in a boiling or substantially boiling sealing bath consisting of the aforesaid mixture in 500 parts by volume of water. The immersion, and concomitant sealing treatment, is continued for about 10 minutes. The resultant sealed, dyed, anodized aluminum does not show any smutty deposit and may be used without supplementary polishing or rubbing.

Example 2

A homogeneous dry mixture is made up by intimately admixing 3.0 parts of nickel acetate crystals, 2.5 parts of boric acid and 2.5 parts of dry desugared calcium lignosulfonate. The mixture is stable on storage for long periods of time.

Example 3

A mixture is made up as per Example 1 or 2, except that the dry desugared calcium lignosulfonate is replaced by a corresponding quantity (about 3.0 parts) of non-desugared calcium lignosulfonate. The mixture is stable on storage, although it is inferior in this respect to the mixture according to the preceding examples.

The mixture is employed after the second paragraph of the latter, whereupon a sealed product is obtained, as precedingly described.

In like manner, another of the hereinbefore-mentioned lignosulfonate molecule-providing materials may be used in lieu of those employed in Examples 1 and 2 and in the first paragraph of the present example, with essentially like results. Where the material is desugared, superior stability on storage is achieved.

The treatment time and temperature of operating set forth in the examples may be varied without appreciably prejudicing the final results.

Having thus disclosed the invention, what is claimed is:

1. In the sealing of dyed anodically oxidized aluminum by means of a hydrolyzable metallic salt in an aqueous bath, the improvement which consists of carrying out the sealing operation in the presence in the bath of at least 0.1% by weight of, as sole sulfonate additament, a compound containing the lignosulfonate radical, whereby a smut-free product which requires no mechanical aftertreatment is obtained.

2. In the sealing of dyed anodically oxidized aluminum by means of a hydrolyzable metallic salt in an aqueous bath, the improvement which consists of carrying out the sealing operation in the presence in the bath of at least 0.1% by weight of calcium lignosulfonate as sole sulfonate additament.

3. In the sealing of dyed anodically oxidized aluminum by means of a hydrolyzable metallic salt in an aqueous bath, the improvement which consists of carrying out the sealing operation in the presence in the bath of at least 0.1% by weight of desugared calcium lignosulfonate as sole sulfonate additament.

4. In the sealing of dyed anodically oxidized aluminum by means of a hydrolyzable metallic salt in an aqueous bath, the improvement which consists of carrying out the sealing operation in the presence in the bath of at least 0.1% by weight of sodium lignosulfonate as sole sulfonate additament.

5. In the sealing of dyed anodically oxidized aluminum by means of a hydrolyzable metallic salt in an aqueous bath, the improvement which consists of carrying out the sealing operation in the presence in the bath of at least 0.1% by weight of dried sulfite waste liquor as sole sulfonate additament.

6. An additive for the production, upon incorporation into water, of a sealing bath for dyed anodically oxidized aluminum, said additive consisting essentially of a dry mixture of substantially equal parts by weight of hydrolyzable metallic salt and a compound containing the lignosulfonate radical, whereby, upon sealing the aluminum in the said bath, a smut-free product which requires no mechanical aftertreatment is obtained.

7. An additive for the production, upon incorporation into water, of a sealing bath for dyed anodically oxidized aluminum, said additive consisting essentially of a dry mixture of substantially equal parts by weight of (*a*) at least one member selected from the group consisting of nickel acetate and cobalt acetate, and of (*b*) calcium lignosulfate.

8. An additive for the production, upon incorporation into water, of a sealing bath for dyed anodically oxidized aluminum, said additive consisting essentially of a dry mixture of substantially equal parts by weight of (*a*) at least one member selected from the group consisting of nickel acetate and cobalt acetate, and of (*b*) desugared calcium lignosulfonate.

9. An additive for the production, upon incorporation into water, of a sealing bath for dyed anodically oxidized aluminum, said additive consisting essentially of a dry mixture of substantially equal parts by weight of (*a*) at least one member selected from the group consisting of nickel acetate and cobalt acetate, and of (*b*) sodium lignosulfonate.

10. An additive for the production, upon incorporation into water, of a sealing bath for dyed anodically oxidized aluminum, said additive consisting essentially of a dry mixture of substantially equal parts by weight of (*a*) at least one member selected from the group consisting of nickel acetate and cobalt acetate, and of (*b*) dried sulfite waste liquor.

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