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PROVISIONAL SPECIFICATION

ELECTRODEPOSITION OF SHELLAC FROM AQUEOUS SYSTEMS

COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH RAFI MARG, NEW DELHI 1, INDIA, AN INDIAN REGISTERED
BODY INCORPORATED UNDER THE REGISTRATION OF SOCIETIES ACT (ACT XXI OF 1860)

The following specification describes the nature of this invention.

This is an invention by Dr. Sankaran Guruswamy, Scientist, Central Electrochemical Research Institute, Karaikudi-3 (Tamil Nadu),
Shri Sthanusubramonea Iyer Krishnamoorthy and Shri Venkataraman Yegnaraman, both Senior Laboratory Assistants,
Central Electrochemical Research Institute Karaikudi-3 (Tamil Nadu), all Indian Citizens.

This invention relates to the electrodeposition of organic coatings from aqueous system on metals. Organic coatings have been extensively used to obtain hard coatings for the protection of metals against corrosion and for electrical insulation. Organic coatings are generally applied to metals from solution/dispersions of organic resins in organic liquids. Organic liquids serve no purpose other than being a convenient medium for the transfer of organic materials on to metals and there is an element of risk of fire in the large scale use of organic liquids.

The object of this invention is to obviate these disadvantages by a) depositing with the help of electric current organic polymers and resins from suitably stabilised aqueous colloidal system of the resins and b) heat treatment of the electrodeposit to modify its properties.

To these ends, the invention broadly consists in a) preparing suitably stabilised aqueous colloidal system of shellac, b) depositing the shellac under definite voltage and current conditions and c) the heat treatment of the deposit in air to modify its properties.

Shellac contains free acid groups which can be partially neutralized by ammonia or other amines such as mono, di and triethanolamines and di and triethyl amines to produce water soluble systems as in the case of linseed oil-maleic acid resin system described in our earlier Indian patent No. 129510. We have found that, if the shellac is initially dissolved in alcohols like isoamyl alcohol, smaller quantities of amines are adequate to produce water dispersions and if small quantities of mercapto benzothiazole were added to the dispersion, heating effects do not appear in the bath at higher voltages; excessive heating effects of the bath are not conducive to the preparation of useful electrodeposit of shellac.

In the typical examples given below, we used isoamyl alcohol for initially dissolving the shellac and triethanolamine for neutralizing the acid groups :

Example 1

50 parts by weight of shellac flakes were dissolved in 110 parts of isoamyl alcohol. 3 parts of a dispersing agent like Swanic 51 (a product of Messrs. Swastik Oil Mills Ltd., Bombay) were gradually added to the solution and dispersed well with the help of a laboratory size (diameter of the vibrator 1") ultrasonic processor. The concentration of the shellac solution was 37%.

54 parts of the shellac solution as above were dispersed in 500 parts of water containing 2% triethanolamine and that dispersion was used for electrodeposition experiments. The electrodes (area of deposition 3.3 x 2.5 cm²) used were mild steel plates kept apart by a distance of 3.2 cm. Direct current was passed through the dispersion at 30 volts for three minutes. The deposit was washed and heat-treated in air at 200°C for one hour.

Nature of the deposit : Reddish black, thick,
smooth, glossy and adherent

Example 2

54 parts of the shellac solution in isoamyl alcohol described in Example I was dispersed in 500 parts of water containing 2% triethanolamine and 0.2% mercapto benzthiazole. Deposition was carried out on mild steel plates using a D.C. voltage of 20 for three minutes. The deposit was washed and heat-treated as in Example I.

Nature of the deposit : Golden yellow, smooth
glossy, transparent, uniform, adherent and satisfactory.

Example 3

Same as Example II. Voltage 200 V D.C. Deposition time and heat treatment same as in Example I.

Nature of the deposit : Reddish black, smooth, opaque,
uniform and adherent ; satisfactory.

The following are the main advantages of the invention :

1. The process has the advantage of speed and the ease of production of organic coatings with the minimum damage done to the metal ;
2. The process is capable of being adapted to the needs of continuous coating of metal articles in commerce and industry.

R. BHASKR PAI.

PATENTS OFFICER

Council of Scientific and Industrial Research.

Dated this 2nd day of March, 1971.

Price : RUPEES TWO.

COMPLETE SPECIFICATION

ELECTRODEPOSITION OF SHELLAC FROM AQUEOUS SYSTEMS.

COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH, RAFI MARG, NEW DELHI-1, INDIA, AN INDIAN REGISTERED BODY INCORPORATED UNDER THE REGISTRATION OF SOCIETIES ACT (ACT XXI OF 1860).

The following specification particularly describes and ascertains the nature of this invention and the manner in which it is to be performed :

This is an invention by Dr. Sankaran Guruswamy, Scientist, Sthanusubramonea Iyer Krishnamoorthy, Senior Laboratory Assistant, and Venkataraman Yegnarman, Senior Laboratory Assistant, all of the Central Electrochemical Research Institute, Karaikudi-3, India, all Indian citizens.

This invention relates to the electrodeposition of organic coatings from aqueous system on metals. Organic coatings have been extensively used to obtain hard coatings for the protection of metals against corrosion and for electrical insulation. Organic coatings are generally applied to metals from solutions/dispersions of organic resins in organic liquids. Organic liquids serve no purpose other than being a convenient medium for the transfer of organic materials on to metals and there is an element of risk of fire in the large scale use of organic liquids.

The object of this invention is to obviate these disadvantages by a) depositing with the help of electric current organic polymers and resins from suitably stabilized aqueous colloidal system of the resins and b) heat treatment of the electrodeposit to modify its properties.

To these ends, the invention broadly consists in a) preparing suitably stabilized aqueous colloidal system of shellac, b) depositing the shellac under definite voltage and current conditions and c) the heat treatment of the deposit in air to modify its properties.

Shellac contains free acid groups which can be partially neutralized by ammonia or other amines such as mono-di and triethanolamines and di and triethylamines to produce water soluble systems as in the case of linseed oil-maleic acid resin system described in our earlier Indian patent No. 129510. We have found that, if the shellac is initially dissolved in alcohols like isoamyl alcohol, smaller quantities of amines are adequate to produce water dispersions and if small quantities of mercapto benzothiazole were added to the dispersion, heating effects do not appear in the bath at higher voltages; excessive heating effects of the bath are not conducive to the preparation of useful electrodeposit of shellac.

According to the present invention, there is provided a process for the preparation of shellac coatings over mild steel, brass and zinc which comprises the covering of the metal by a film of shellac, wherein the shellac is solubilised in water into a colloidal system by dissolving in isoamyl alcohol by gentle heating and neutralising the acid groups of the shellac by triethanolamine, and the colloidal shellac is electrodeposited by using a direct current in the voltage range 20 to 200 volts, and the properties of the deposited shellac modified by the heat-treatment of the deposit in air in the temperature range of 120 to 260°C. for a period of 30 to 100 minutes.

We have found that deposits in different colour shades in the colour range golden yellow—pale green—brown—reddish brown—black can be prepared by using different metals and by using different grade shellac samples and by varying the electrical conditions of deposition and also by varying the time and temperature at which the shellac deposits are baked. We have also found that by adding bitumen to shellac, we can prepare glossy black deposits over metals by baking the deposits at comparatively lower temperatures.

In the typical examples given below, we used isoamyl alcohol for initially dissolving the shellac and triethanolamine for neutralizing the acid groups :

Example I

50 parts by weight of D.L. shellac flakes were dissolved in 110 parts of isoamyl alcohol. 3 parts of a dispersing agent like Swanic 51 (a product of Messrs. Swastik Oil Mills Ltd., Bombay) were gradually added to the solution and dispersed well with the help of a laboratory size (diameter of the vibrator $\frac{1}{4}$ ") ultrasonic processor. The concentration of the shellac solution was 37%

54 parts of the shellac solution as above were dispersed in 500 parts of water containing 2% triethanolamine and that dispersion was used for electrodeposition experiments. The electrodes (area of deposition $3.3 \times 2.5 \text{ cm}^2$) used were mild steel plates kept apart by a distance of 3.2 cm. Direct current was passed through the dispersion at 30 volts for three minutes. The deposit was washed and heat treated in air at 200°C for one hour. Deposition takes place on the anode.

Nature of the deposit : Reddish black, thick, smooth, glossy and adherent

Example II

54 parts of the shellac solution in isoamyl alcohol described in Example I was dispersed in 500 parts of water containing 2% triethanolamine and 0.2% mercapto benzothiazole. Deposition was carried out on mild steel plates using a D.C. voltage of 20 for three minutes. The deposit was washed and heat treated as in Example I. Deposition takes place on the anode.

Nature of the deposit : Golden yellow, smooth glossy, transparent, uniform, adherent and satisfactory

Example III

Same as Example II. Voltage 200 D.C. Deposition time and heat treatment same as in Example I. Deposition takes place on the anode.

Nature of the deposit : Reddish black, smooth, opaque, uniform and adherent : satisfactory

Example IV

35 parts of hydrolysed shellac (BOL grade) is dissolved in 40 parts of isoamyl alcohol by slow heating to obtain a uniform resinous mass. 8 parts of the above resin are dispersed and solubilized in 100 parts of water containing 0.8% of triethanolamine to form an opaque emulsion which is used in the electrodeposition experiments. Brass plates (area of deposition $3.3 \times 2.5 \text{ cm}^2$) were used as anodes and the cathode was a mild steel plate of the same size. Direct current was passed through the electrodes kept at a distance of 5 cms. at 100 volts for three minutes. The electrodeposits over brass were baked in

the temperature range of 120 to 240°C for a period of three hours to 45 minutes to obtain coatings in the colour range pale green = brown — reddish brown — black.

Example V

The electrodeposition bath was prepared as in Example IV. Zinc plates were used as anodes and all other deposition conditions were the same as in Example IV except for the voltage which was maintained at 50V. Electrodeposits over zinc were cured in the temperature range 180° to 260°C for one hour to obtain coatings in the colour range pale greylight brown—brown—black.

Example VI

8 parts of the resinous mass prepared as in Example IV was slowly heated with 1.5 parts of bitumen to obtain a homogeneous black viscous mass. To the cooled mass was added 0.8 parts of triethanolamine and the entire mass is solubilized in 100 parts of water to obtain a suspension. The suspension is boiled and then filtered to get a dark brown opaque emulsion for electrodeposition experiments. Using a direct current of 100 volts for three minutes, smooth glossy adherent black coatings over brass plate anodes were obtained by baking the electrodeposits in the temperature range of 130 to 200°C over a period of 100 to 30 minutes.

Example VII

Applying a D.C. voltage in the range 50 to 100, smooth, adherent, semi-glossy black deposits over mild steel were prepared from electrodeposition bath as in Example VI; heat treatment being the same as in Example VI.

Example VIII

Using a D.C. voltage of 75 in the electro bath as in Example VI, semiglossy, brown-reddish brown-black coatings over zinc were prepared by baking the electrodeposits in the temperature range of 120 to 220°C for a period of 100 to 50 minutes.

The following are the main advantages of the invention;

1. The process has the advantage of speed and ease of production of organic coatings with the minimum damage to the metal.

2. The process is capable of being adapted to the needs of continuous coating of metal articles in commerce and industry.

In this Patent, electrocoatings of shellac over mild steel brass and zinc are prepared from suitably dispersed and stabilized aqueous systems of shellac. For solubilizing the shellac, alcohols like isoamyl alcohol and bases like triethanolamine are used. Chemicals like mercapto benzothiazole are added to overcome the heating effects in the bath which effects appear when shellac flakes are used for dispersion, and when the deposition is carried out at higher voltages. We have also found that with the addition of bitumen to shellac, glossy black deposits can be prepared over metals by baking the deposits at comparatively lower temperatures.

We claim :

1. A process for the preparation of shellac coatings over mild steel, brass and zinc which comprises the covering of the metal by a film of shellac, wherein the shellac is solubilised in water into a colloidal system by dissolving in isoamyl alcohol by gentle heating and neutralising the acid groups of the shellac by triethanolamine, and the colloidal shellac is electrodeposited by using a direct current in the voltage range 20 to 200 volts and the properties of the deposited shellac modified by the heat-treatment of the deposit in air in the temperature range of 120 to 260°C. for a period of 30 to 100 minutes.

2. A process as claimed in 1 wherein the addition of 0.2% mercapto benzothiazole enables to overcome heating effects of the aqueous shellac electrocoating bath at higher voltages

3. A process as claimed in 1 wherein the addition of bitumen to the shellac electrocoating bath enables the preparation of glossy black deposits over metals by baking the deposits at comparatively lower temperatures.

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Council of Scientific & Industrial Research

Dated this 4th day of January, 1972