GOVERNMENT OF INDIA: THE PATENT OFFICE, 214, ACHARYA JAGADISH BOSE ROAD, CALCUTTA-17.

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ELECTRODEPOSITION OF CASTOR OIL MALEIC ANHYDRIDE RESINS ON MILD STEEL.

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, India and Shri Pokkyarath Jayakrishan, Junior Scientific Assistant, Central Electrochemical Research Institute, Karaikudi-3, (Tamil Nadu), India, both Indian citizens.

This invention relates to the electrodeposition of caster oil maleic anhydride resins on mild steel. Hitherto it has been proposed to obtain organic coatings for the protection of metals against corrosion and for electrical insulation by dipping the metal in a solution of the resin dissolved in organic solvents. This is open to objection in that organic solvents in the present context serve no purpose other than being a convenient medium for the transfer of organic materials on to metals. There is further an element of risk of fire in the large scale use of organic solvents.

The object of this invention is to obviate these disadvantages by (a) depositing with the help of direct electric current organic resin prepared from castor oil maleic anhydride from suitably stabilised aqueous colloidal system of the resins and (b) heat treating of the electrodeposit to modify its properties.

To these ends, the invention broadly consists in (a) preparing suitably stabilised aqueous colloidal system of the castor oil maleic anhydride resin, (b) depositing the said resin under a D. C. voltage of 20 to 100 volts and (c) the heat treatment of the deposit in the temperature of 150 to 200°C to modify its properties.

In our experiments, castor oil was reacted with maleic anhydride to obtain water dispersible/soluble adduct. Alcohols similar to propylene glycol and amines similar to triethanolamine were added to aid and increase the solubilization and neutralisation respectively of the resin in water. The pH of the system was carefully controlled by suitable additions of the amines. The right conditions for deposition were obtained by controlling the voltage of the deposition and the current density. The right shade of colour of the coating within the range of yellow-red-black, was obtained by controlling the pH of the bath, baking time and temperature of the oven.

The following typical examples are given to illustrate the invention :

Example 1

The preparation of an electrodeposit having high corrosion resistance which can stand 56 hours (7 days of 8 hours duration per day) of salt syray test and having an electrical breakdown voltage of 3000 (DC) and insulation resistance of 525 mega ohms is as follows :

83 parts of commercial quality castor oil are heated with 40 parts of maleic anhydride and 17 parts of diethyl pthalate at a temperature of 200-220°C for a period of 30 minutes and cooled to form a viscous resin. 2.5 gms. of the resin is solubilized in 50 ccs. of water containing 1.25 ml. of triethanolamine to give 5% resin and amine/resin ratio of 0.5. 0.25 gms. of 2-Mercaptobenzothiazole was dissolved in the dispersion. The electrodeposits were prepared on mild steel plates of nominal size 4″×1″ kept at a distance of 3.2 cms. The area of the deposition was 3.3×2.5 sq. cm. and the cathode area was the same as the anode area. The voltage applied was 30 (DC) and the duration of the deposition was three minutes. The temperature of baking was between 180-200°C.

Example 2

The preparation of an electrodeposit having high corrosion resistance, i.e. 56 hours of salt spray test as mentioned in Example 1, and having a breakdown voltage of 1000 (DC) was carried out by taking 3 gms. of the resin as prepared in Example 1, solubilized in 50 ccs. of water containing 1.5 gms. of amine and 0.5 gms. of 2-Mercaptobenzothiazole. The electrodeposits were prepared on mild steel plates of dimensions given in Example 1 at a D.C. voltage of 100. The time of deposition and the temperature of baking are the same as given in Example 1.

Example 3

A high glossy deposit is prepared by solubilising 2.5 gms. of resin. 1.25 gms. of amine and adding 5-10% of propylene glycol to bring the final volume to 50 cc. Voltage, the time of deposition and the temperature of baking are the same as given in Example 1.

It can generally be stated that deposits are in the colour range yellow-red black and tend to become glossier and smoother as the temperature of baking is increased from 180°C and the time of baking at higher temperatures (upto 200°C) is correspondingly reduced.

The following are among the main advantages of the invention :

1. The process has the advantages of ease of production of organic coatings with minimum damage to the underlying metal.

2. The process is capable of being adapted to the needs of continuous coating of mild steel articles of industry and commerce, which need organic coatings for corrosion protection and electric insulation.

Dated this 8th day of July, 1971.

Sd

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Price : TWO RUPEES.

COMPLETE SPECIFICATION

ELECTRODEPOSITION OF CASTOR OIL MALEIC ANHYDRIDE RESINS ON MILD STEEL

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)

THIS IS AN INVENTION BY DR. SANKARAN GURUSWAMI, SCIENTIST AND POKKYARTH JAYAKRISHNAN, JUNIOR SCIENTIFIC ASSISTANT, BOTH OF THE CENTRAL ELOCTROCHEMICAL RESEARCH INSTITUTE, KARAIKUDI 25, TAMIL NADU, INDIA, BOTH INDIAN CITIZENS.

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

The invention relates more particularly to the electrodeposition of resins from aqueous systems and has got its principal object to prepare electro-organic coatings from castor oil-maleic anhydride resins on mild steel.

It is further the object of this invention that it should be possible to modify the properties of the electrodeposits by incorporating resin and shellac into the castor oil-maleic anhydride resin system.

Hitherto it has been proposed to obtain organic coatings for the protection of metals against corrosion and for electrical insulation by dipping the metal in a solution of the resin dissolved in an organic solvent. This is open to objection in that organic solvents in the present context serve no purpose other than being a convenient medium for the transfer of organic materials on to metals. There is further an element of risk of fire in the large scale use of organic solvents.

The main object of this invention is to obviate these disadvantages by (a) depositing with the help of electric current organic resin prepared from castor oil-maleic anhydride from suitably stabilized aqueous colloidal system of the resin and (b) heat-treating the electrodeposits to modify its properties.

It is further the object of this invention to modify further the properties of these deposits by suitably incorporating into the resin measured quantities of shellac and resin in order to modify the properties of the deposits.

According to the present invention, there is provided a process for the electrodeposition of castor oil-maleic anhydride resins on mild steel which consists in heating castor oil with maleic anhydride, resin, shellac, isoamyl alcohol, the resultant adduct is water-solubilized by the partial neutralization of the acid groups in the adduct by a base to yield a negatively charged poly-electrolyte of the resin in water which is deposited on mild steel anode by the passage of current and the electrodeposit thus obtained is baked to develop colour and adhesion.

Propylene glycol is added to aid the water solubility of the resin and develop and control the glossiness of the deposit.

Amines like triethanolamine are added to neutralize the acid groups in the resin and disperse and solubilize the castor oil-maleic anhydride resin.

The rosin and shellac are added in 7 to 25% concentration to develop and control the adhesion of the deposit.

The isoamyl alcohol in concentration of 10 to 30% is added to incorporate larger amounts of shellac into the castor oil-maleic anhydride resin.

The resin is deposited over mild steel articles by covering of the metal article by an organic film former by the following steps:

(a) preparing a suitably stabilized aqueous colloidal system of castor oil-maleic anhydride resin with and without the addition of rosin and shellac;

(b) depositing the said resin by electric current;

(c) heat-treating of the deposit at a temperature of 150-200°C to develop colour and adhesion.

The depositing is done with the help of alternating as well as direct electric current.

The steps are:

(a) preparation of water soluble resins suitable for electrodeposition;

(b) depositing the resin over mild steel by electric current;

(c) baking the electrodeposit to develop colour and adhesion.

To these ends, the invention broadly consists in (a) preparing a suitably stabilized aqueous colloidal system of castor oil-maleic anhydride resin with and without the addition of rosin and shellac, (b) depositing the said resin by electric current and (c) heat-treating of the deposit at a temperature of 150-200°C to modify its properties.

In our experiments, castor oil was reacted with maleic anhydride to obtain a water dispersible soluble adduct. Alcohols similar to propylene glycol and amines similar to triethanolamine were added to aid and increase solubilization and neutralisation respectively of the resin in water. The pH of the system was carefully controlled by suitable additions of the amines. The right conditions for deposition were obtained by controlling the voltage of deposition and current density. The right shade of the colour of the coating within the range yellow-reddish brown-black was obtained by controlling the pH of the bath, baking time and temperature.

The present invention consists of a process for the preparation of electro-organic coatings from maleinised castor oil over mild steel which comprises the covering of the metal by a film of the said resin wherein the resin is solubilized in water in the colloidal system and the said colloidal resin is electrodeposited under a definite voltage and current conditions and the properties of the deposited resin modified by heat-treatment of the electrodeposit in air. We have found that the deposits in different colour shades yellow-reddish, brown-black can be prepared over mild steel by varying the electrical conditions of the deposition and also by varying the time and temperature, at which the electrodeposits are baked. We have also found that by adding shellac and rosin in measured quantities we can control the glossiness and adhesion of the electrodeposits over mild steel.

The following typical examples are given to illustrate the invention:

Example 1

The preparation of an electrodeposit having high corrosion resistance which can stand 56 hours (7 days of 8 hours duration per day) of salt spray test and having an electrical breakdown voltage of 3000 (d.c.) and insulation resistance of 525 mega ohms is as follows:

83 parts of commercial quality castor oil are heated with 40 parts of maleic anhydride and 17 parts of diethyl pthalate at a temperature of 200-220°C for

a period of 30 minutes and cooled to form a viscous resin. 2.5 gms. of the resin is solubilized in 50 ccs. of water containing 1.25 ml. of triethanolamine to give 5% resin and amine/resin ratio of 0.5. 0.25 gms. of 2-mercaptobenzothiazole was dissolved in the dispersion. The electrodeposits were prepared on mild steel plates of nominal size 4"×1" kept at a distance of 3.2 cms. The area of the deposition was 3.3×2.5 sq. cm. and the cathode area was the same as the anode area. The voltage applied was 30 (d.c.) and the duration of the deposition was three minutes. The temperature of baking was between 180-200°C.

Example 2

The preparation of an electrodeposit having high corrosion resistance, i.e. 56 hours of salt spray test as mentioned in Example 1, and having a breakdown voltage of 1000 (d.c.) was carried out by taking 3 gms. of the resin as prepared in Example 1, solubilized in 50 ccs. of water containing 1.5 gms. of amine and 0.5 gm. of 2-mercaptobenzothiazole. The electrodeposits were prepared on mild steel plates of dimensions given in Example 1 at a d.c. voltage of 100. The time of deposition and the temperature of baking are the same as given in Example 1.

Example 3

A high glossy deposit is prepared by solubilizing 2.5 gms. of resin, 1.25 gms. of amine and adding 5 to 10% of propylene glycol to bring the final volume to 50 cc. Voltage, the time of deposition and the temperature of baking are the same as given in Example 1.

It can generally be stated that deposits are in the colour range yellow-red black and tend to become glossier and smoother as the temperature of baking is increased from 180°C and the time of baking at higher temperatures (upto 200°C) is correspondingly reduced.

Example 4

The preparation of an electrodeposit having very high gloss has been carried out by taking ten parts by weight of the resin as prepared in Example 1 and incorporating into it 0.1 to 0.5 parts of hydrolysed shellac (BOL grade) at a temperature of 200°C. 5 parts of the resin as prepared above is solubilized in 100 parts of water by the addition of triethanolamine to bring the pH to 7. The electrodeposits were prepared on mild steel plates of dimensions given in Example 1 at a.c. voltage of 220 using for one of the electrodes aluminium wire of diameter about 1mm. The time of deposition was one minute. The deposits were baked at a temperature of 180°C for a period of 30 minutes.

Example 5

The preparation of a dull deposit having good adhesion was carried out by treating 10 parts of the resin as in Example 1 with 1 to 2 parts of shellac and 5 parts of isoamyl alcohol at 200°C for a period of five minutes. The resultant homogeneous product was solubilized in water by adding triethanolamine and electrodeposition carried out at a pH of 7 at a d.c. voltage of 50. The time of deposition was three minutes and the temperature and time of baking the same as in Exmaple 4.

Example 6

Good glossy adherent deposits were prepared by incorporating 2 parts of rosin into 10 parts of the resin prepared as in Example 1, at 200°C and solubilizing the resultant product by triethanolamine to a pH of 7. The voltage and time of deposition and also time and temperature of baking are the same as given in Example 4.

It can generally be stated that the deposits are in the colour range yellow-reddish, brown-black and tend to become glossier and smoother as the temperature of baking is increased from 180°C and the time of baking at higher temperature, i.e. upto 200°C., is correspondingly reduced.

The following are the main advantages of the invention :

1. The process has the advantage of ease of production of organic coating with minimum damage to the underlying metal.

2. The process is capable of being adapted to the needs of continuous coating of mild steel articles of industry and commerce which need the organic coatings for corrosion resistance and electrical insulation.

Baked electrodeposits from maleinised castor oil resin have been prepared by electrodepositing the resin on the mild steel articles to be coated by the passage of electric current through a bath containing an aqueous colloidal system of the resin and baking the electrodeposit to develop colour and adhesion. The aqueous system is prepared by solubilizing the maleinised raw castor oil by partial neutralisation of the acid groups by amines like triethanolamine. Alcohols like propylene glycol and materials like mercaptobenzothiazole, rosin and shellac are incorporated in the resin to develop and control the glossiness and adherence of the electrodeposits.

WE CLAIM :

1. A process for the electrodeposition of castor oil-maleic anhydride resins on mild steel which consists in heating castor oil with maleic anhydride, rosin, shellac, isoamyl alcohol, the resultant adduct is water-solubilized by the partial neutralization of the acid groups in the adduct by a base to yield a negatively charged polyelectrolyte of the resin in water which is deposited on mild steel anode by passage of current and the electrodeposit thus obtained is baked to develop colour and adhesion.

2. A process as claimed in Claim 1 wherein propylene glycol is added to aid the water solubility of the resin and develop and control the glossiness of the deposit.

3. A process as claimed in Claim 1 or 2 wherein amines like triethanolamine are added to neutralize the acid groups in the resin and thereby disperse and solubilize the castor oil-maleic anhydride resin.

4. A process as claimed in any of the preceding claims wherein rosin and shellac are added in 7 to 25% concentration to develop and control the adhesion of the deposit.

5. A process as claimed in any of the preceding claims wherein isoamyl alcohol is added in concentration of 10 to 30% to incorporate larger amounts of shellac into the castor oil-maleic anhydride resin.

6. A process as claimed in any of the preceding claims wherein the resin is deposited over mild steel articles by covering the metal article by an organic film former by the following steps :

(a) preparing a suitably stabilized aqueous colloidal system of castor oil-maleic anhydride resin with and without the addition of rosin and shellac ;

(b) depositing the said resin by electric current ;

(c) heat-treating of the deposit at a temperature of 150-200°C to develop colour and adhesion.

7. A process as claimed in Claim 6 wherein depositing is done with the help of the alternaing as well as direct electric current.

8. A process for the electrodeposition of castor oil-maleic anhydride resins on mild steel articles substantially as hereinbefore described.

9. Mild steel articles on which castor oil-maleic anhydride resins have been deposited according to a process substantially as hereinbefore described.

Dated this 16th day of May 1972.

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