Government of India, The Patent Office, 214, Acharya Jagadish Bose Road, Calcutta-17. Complete specification No.141458 dated 19th February 1975. Application and Provisional specification No.346/Cal/1974 dated 19th February 1974. Acceptance of the complete specification advertised on 5th March 1977.

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METHOD OF PRODUCING AND SEALING COLOURED ALUMINIUM POWDERS

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 xxx this invention.~~

This is an invention by BALKUNJE ANANTHA SHENOI, Scientist, and SUBBIAH JOHN, Junior Scientific ~~xx~~ Assistant, both are Indian Nationals and employed in the Central Electrochemical Research Institute, Karaikudi-623003, Tamil Nadu, India.

The following specification describes the nature of this invention.

PRICE, RS. 2-00

This invention relates to methods of sealing coloured aluminium powders.

Sealing has been defined as a process which by absorption, chemical reaction, or other mechanism increases the resistance of an oxide coating to staining and to corrosion, improves the durability of colour produced in the coating or imparts other desirable properties. The coloured aluminium powders are widely used in the printing trades for printing on textiles, leather and paper. In the textile printing field, the exacting requirement of the coloured aluminium powder is that the colour should not fade when washed with detergents. The life, appearance and corrosion resistance of the oxide coating are influenced by the sealing process.

Aluminium powder is coloured by producing an oxide coating by chemical conversion coating means to form a base for organic finishing. The thinner and softer oxide films are coloured with organic dyes to produce the aesthetic appearance. In order to fix the dye in the oxide coating and to prevent bleeding while washing, it is necessary to seal the coating in suitable sealants.

The main object of the present invention is to find out a suitable sealant for the sealing the coloured aluminium powders. It is found that by incorporating suitable proprietar addition agents along with the known sealants for anodic oxide coatings, the physical characteristics of the cploured powder is improved upon sealing in such a solution.

The main finding underlying the invention consists in sealing the coloured aluminium powder having different mesh sizes in a bath consisting of the following chemicals:(1) Heavy metal acetate, (2) Mannitol, (3) Lignosol, (4) Phthalic anhydride and (5) Distilled water at a temperature of 90-100°C for 15-20 minutes.

The chemically oxidised powder is filtered, washed and then coloured with conventional acid dyes. The colour and concentration of the dyes can be varied according to the requirements. Finally, the coloured powder is sealed in the following manner.

EXAMPLE 1

MS 7508 aluminium powder having -100 to +150 BS mesh size is oxidised according to the Indian Patent Application No.374/Cal/73. It is then washed and dyed in the following dye bath:

Dye : Keviluminium Gold Yellow LL	
Dye concentration:	7g/100g powder
pH	6 ± 0.5
Dye bath temperature	60-65°C
Dyeing time	15-25 minutes

The dyed powder is filtered and transferred to the sealing bath. Sealing condition is as follows :

Nickel acetate:	15 g/l
Cobalt acetate:	3 g/l
Mannitol:	0.2 g/l
Lignosol	0.5 g/l

Phthalic anhydride:	0.5 g/l
Sealing temperature:	90-100°C
pH	5.5 ± 0.5
Sealing time:	15-20 minutes

The sealed powder is filtered, washed and then dried in an air oven at a temperature of 70-80°C for 1-2 hours. There is no bleeding of the dye during the sealing treatment and hence the colour of the sealed powder is good. The sealed powder is very good for textile printing.

EXAMPLE 2

MS 7508 aluminium powder having -100 to +150 BS mesh size is oxidised and after washing it is dyed under the following conditions:

Dye: Durand & Huguenin Red

Dye concentration:	7g/100g powder
Dye bath pH	5.5 ± 0.5
Dyeing temperature	60-65°C
Dyeing time:	15-25 minutes

After dyeing, it is filtered and transferred to the sealing bath. The sealing condition is as follows:

Lead acetate	10 g/l
Mannitol	0.2 g/l
Lignosol	0.5 g/l
Phthalic anhydride	0.5 g/l
Sealant pH	5.5 - 6.0
Sealing time	15-20 minutes
Sealing temperature	95-100°C

The sealed powder is filtered, washed and then dried. There is no bleeding of the dye during the sealing treatment and hence the colour of the powder is good. The sealed powder is very good for paper printing.

EXAMPLE 3

MS 7512 aluminium powder having -120 to 200 BS mesh size is oxidised according to method mentioned in Example 1. It is then filtered and dyed in the following dye bath:

Dye: CIBA Violet dye	
Dye concentration:	6.5 g/100g powder
Dye bath pH	4.5-5.0
Temperature of dye bath	60-65°C
Dyeing time	15-25 minutes

The dyed powder is filtered and transferred to the sealing bath. The sealant composition and operating conditions are the same as in Example 2. The colour of the sealed powder is very attractive and hence suitable for textile and paper printing fields.

EXAMPLE 4

MS 7508 aluminium powder having -100 to + 150BS mesh size is oxidised, filtered and washed. It is dyed under the following conditions:

Dye : Devar Anodising Copper Red Madhavdas Manilal & Co.	
Dye concentration:	7g/100g powder
Dye bath pH	6.0 ± 0.5
Temperature of dye bath:	60-65°C
Dyeing time:	15 - 25 minutes

The dyed powder is filtered and transferred to the sealing bath. The sealant composition is as given in Example 2.

There is no bleeding at all during and after the sealing treatment and hence the colour of the powder is excellent. The sealed powder is very good for printing on textiles and paper and are suitable for preparing enamel paints.

The following are among the main advantages of the invention:

1. The chemicals involved in this process are indigenously available.
2. This coloured and sealed powders can be used as a substitute for bronze powder which is used by the printing industries for printing on textiles, paper and leather.
3. This can be used even in manufacturing aluminium enamel paints with different colours.
4. The sealed powder is resistant to soap water wash and hence there is no bleeding while washing. This physical characteristic makes it suitable for printing on textiles.

Dated this 15th day of February, 1974

Asstt. Patents Officer,
Council of Scientific & Industrial Research

THE PATENTS ACT 1970

COMPLETE SPECIFICATION

SECTION 10

METHOD OF PRODUCING AND SEALING COLORED

ALUMINIUM POWDERS

COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH, Rafi Marg, New Delhi-1, India, an Indian registered body incorporated under the Registration of Societies Act (ACT XXI of 1860).

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This is an invention by BALKUNJE ANANTHA SHENOI, Scientist and SUBHAIAH JOHN, Junior Scientific Assistant, both are Indian Nationals and employed in the Central Electrochemical Research Institute, Karaikudi-623 006, Tamil Nadu, India.

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

This invention relates to method of Producing and sealing coloured aluminium powders.

Sealing has been defined as a process which by absorption, chemical reaction, or other mechanism increases the resistance of an oxide coating to staining and to corrosion, improves the durability of colour produced in the coating or imparts other desirable properties. These coloured aluminium powders are widely used in the printing trades for printing on textiles, leather and paper. In the textile printing field, the exacting requirement of the coloured aluminium powder is that the colour should not fade when washed with detergents. The life, appearance and corrosion resistance of the oxide coating are influenced by the sealing process.

Since colouring of aluminium powder by chemical methods is a new process, there is no information available regarding sealing of this coloured aluminium powder. Hence, there is no prior knowledge available about the hitherto known processes.

Aluminium powder is coloured by producing an oxide coating by chemical conversion coating means to form a base for organic finishes. The thinner and softer oxide films are coloured with organic dyes to produce the aesthetic appearance. In order to fix the dye in the oxide coating and to prevent bleeding while washing, it is necessary to seal the coating with suitable sealants.

The main object of the present invention is to find out suitable sealants for the sealing of chemically oxidised and dyed aluminium powders of different colours.

The main finding underlying the invention consists in sealing the coloured aluminium powders in a bath comprising the following chemicals:

1) heavy metal acetate 2) Polyhydric alcohol
3) phthalic anhydride 4) lignosol and
5) distilled water

The new result flowing from the new finding is that dyed and sealed powders printed on textiles do not bleed upon washing with soap and hence there is no colour change of the textiles and the colour of the powder does not fade upon exposure to the Sun. It is found that the light fastness of the powder is the same as that of the textile upon which it is printed.

According to the present invention, there is provided a process for producing and sealing coloured aluminium powder of different mesh size which comprises the steps of degreasing, chemical oxidising in an aqueous alcoholic solution consisting

of methanol, sodium hydroxide, sodium salicylate, sodium aluminate and methanol at temps of 35-60°C for 30 to 180 min. and subsequently colouring the oxidised powder in aqueous acidic organic dye solutions and finally sealing the coloured powder in an aqueous sealing bath containing heavy metal acetates, polyhydric alcohol, lignosol and phthalic anhydride at temps of 90-100°C for 15 min at a pH of 5.0 to 6.0.

Sodium succinate and sodium oxalate may also be added to the chemical oxidising bath.

The chemical oxidising bath may consist of methanol 30-60%, sodium hydroxide 0.5-3% sodium aluminate 1.0-2.5%. sodium succinate 1.5%, sodium oxalate 2% and sodium salicylate 1.5%.

The aqueous anodal organic acidic dye solution may consist of 0.01-1% anodal dyes at temp. of 50-60° for 5-20 min. at pH of 5-6.

The sealing bath may consist of heavy metal acetate/s 0.5 - 2.0%, poly hydric alcohol 0.005-0.1%, lignosol 0.01-0.2% and phthalic anhydride 0.01-0.2%.

The heavy metal acetate/s in the sealing bath is/are lead, nickel and cobalt.

The poly hydric alcohol used in the sealing bath is mannitol or sorbitol.

The steps comprise degreasing, chemical oxidising in an aqueous alcoholic solution consisting of 30-60% methanol, 0.5-3% sodium hydroxide, 1.0-2.5% sodium aluminate, 1.5% sodium succinate, 2% sodium oxalate and 1.5% sodium salicylate at temps of 35-60°C for 30-180 min. and subsequently colouring the oxidised powder in 0.01-1% aqueous anodal acidic organic

dyes at temps of 50-60°C for 5-20 min. at pH of 5-6 and finally sealing the coloured powder in an aqueous sealing bath consists of 0.5-2.0% heavy metal acetate, 0.005-0.1% polyhydric alcohol 0.01-0.2% lignosol and 0.01-0.2% phthalic anhydride at temps of 90-100°C for 15 min. at a pH of 5.0 to 6.0 with this process minimum bleaching during sealing and improved resistance for washing can be achieved.

The novel features of this sealing process is that these coloured powders can be used in the printing fields such as textile, leather and paper printing. It has also found important uses in other arts such as for use in lithographic inks, in sign painting, in rubber compounding and in the application of colouring the pigment.

Flowsheet of the process is attached herewith.

The chemically oxidised powder is filtered, washed and then coloured with acidic anodal organic dyes. The colour and concentration of the dyes can be varied according to the requirements. Finally, the coloured powder is sealed. Specific sealants are to be used for each colour.

The following typical examples are given how the invention is carried out in actual practice but not to limit the scope of this invention:

EXAMPLE-1

200 g of M.S. 7508 aluminium powder (minimum 99.6% Al) having -100 to +150 BS mesh size is degreased with acetone and dried at room temp. The dried powder is stirred vigorously in one litre of the bath which is prepared from the following chemicals:

Methanol	..	55%
Sodium hydroxide	..	2%
Sodium aluminate	..	1%
Sodium salicylate	.	1.5%
Distilled water	..	Remaining %
Temp.	..	60°C
Time	..	120 min

The oxidised powder is filtered, washed three or four times with tap water and then with distilled water. The filtered powder is transferred to the dyeing bath which is prepared as follows:

Kevilluminium Anodal Gold yellow LL	.. 0.7%
pH	.. 5.5 ± 0.5
Dyebath temp.	.. 60-65°C
Dyeing time	.. 15-25 min

After colouring the powder is filtered and taken to the following sealing bath:

Nickel acetate	.. 1.5%
Cobalt acetate	.. 0.3%
Mannitol or Sorbitol	.. 0.02%
Lignosol	.. 0.05%
Phthalic anhydride	.. 0.05%
Temp.	.. 90-100°C
pH	.. 5.5 ± 0.5
Time	.. 15 min.

After sealing the powder is filtered, washed and then dried in an air oven at a temp. of 60-70°C for 1 to 2 hrs. There is no bleeding of the dye during the sealing process which normally occur with conventional sealing process. Hence the Colour of the sealed powder is very much attractive and is golden yellow. The powder is best suited for textile printing.

EXAMPLE-II

100 g. of MS 7508 aluminium powder (minimum 99.6% Al) having -120 to 200 B Smesh size is degreased in acetone and dried at room temp. The dried powder is stirred well in one lit. of the bath which is prepared with the following chemicals:

Methanol	..	60%
Sodium oxalate	..	2%
Sodium succinate	.	0.5%
Distilled water	..	remaining %
Temp.	..	45°C
Time	..	120 min.

The oxidised powder is filtered, washed three or four times with tap water and then with distilled water. The filtered powder is transferred to the dye bath which is prepared as follows:

Durand and Huguenin Anodal Red	..	0.6%
Temp.	..	60-65°C
pH	..	5.5 ± 0.5
Time	..	15-25 min.

After colouring it is filtered and transferred to the sealing bath. The sealing condition is as follows:

Lead acetate	..	1.0%
Mannitol or Sorbitol	..	0.02%
Lignosol	..	0.05%
Phthalic anhydride	..	0.05%
pH	..	5.5-6.0
Time	..	15-20 min.
Temp.	..	90-100°C

The sealed powder is filtered, washed and then dried. There is no bleeding of the dye during sealing treatment and hence the colour of the powder is good. The sealed red coloured powder is suitable for paper printing.

EXAMPLE-III

MS7512 aluminium powder (minimum 99.6% Al) having -120 to 200 B Smesh size is chemically oxidised according to method mentioned in Example-I. It is then filtered and dyed in the following dye bath:

CIBA Anodal Violet dye	..	0.65%
pH	..	5.5 ± 0.5
Temp.	..	60 - 65°C
Time	..	20 min.

The coloured powder is filtered and transferred to the sealing bath mentioned in Example-II. The colour of the sealed powder is attractive and is suitable for textile and paper printing.

EXAMPLE-IV

MS 7508 aluminium powder having -100 to +150 B Smesh size is chemically oxidised according to method mentioned in Example-II. It is then filtered and dyed under the following conditions:

Dever Anodising Copper Red	..	0.7%
pH	..	5.5 ± 0.5
Temp.	..	60-65°C
Time	..	10 Min.

The coloured powder is filtered and transferred to the sealing bath mentioned in Example-II.

There is no bleeding at all during and after the sealing treatment and hence the colour of the powder is good. The sealed powder is best suited for printing on textiles and paper, and also suitable for preparing enamel paints.

The following are among the main advantages of this invention:

1. The chemicals involved in this process are easily available in our country.
2. This coloured and sealed powders can be used as substitute for bronze powder which is used by the printing industries for printing on textiles, paper and leather.
3. This can be used even in manufacturing aluminium enamel paints with different colours.

4. The sealed powder is resistant to soap water wash and hence there is no bleeding while washing. This physical characteristic makes it suitable for printing on textiles.

This invention provides a method of producing and sealing coloured aluminium powders for use in the printing industries such as textile, paper and leather. The aluminium powder is degreased, oxidised, coloured with organic dyestuffs and sealed in an aqueous bath containing heavy metal acetate/s, polyhydric alcohol, phthalic anhydride and lignosol at a PH of 5-6 for 15-25 minutes at a temperature of 90-100°C to improve the light fastness and resistance for bleeding.

WE CLAIM

1. A process for producing and sealing coloured aluminium powder of different mesh size which comprises the steps of degreasing, chemical oxidising in an aqueous alcoholic solution consisting of methanol, sodium hydroxide, sodium salicylate and sodium aluminate at temps of 35-60°C for 30 to 80 min. and subsequently colouring the oxidised powder in aqueous acidic organic dye solutions and finally sealing the coloured powder in an aqueous sealing bath containing heavy metal acetates, polyhydric alcohol, lignosol and phthalic anhydride at temps. of 90-100°C for 15 min.at a pH of 5.0 to 6.0

2. A process as claimed in Claim 1 wherein sodium succinate and sodium oxalate are added to the chemical oxidising bath.

3. A process as claimed in Claim 2 wherein the chemical oxidising bath consists of methanol 30-60%, sodium hydroxide 0.5-3%, Sodium aluminate 1.0-2.5%, Sodium succinate 1.5%, Sodium oxalate 2% and Sodium salicylate 1.5%.

4. A process as claimed any of the preceding claims wherein the aqueous organic acidic dye solution consists of 0.01-1% anodal dyes at temp. of 50-60° for 5-20 min. at pH of 5-6.

5. A process as claimed in any of the preceding claims wherein the sealing bath consists of heavy metal acetate/s 0.5 - 2.0%, polyhydric alcohol 0.005-0.1%, lignosol 0.01-0.2% and phthalic anhydride 0.01-0.2%

6. A process as claimed in any of the preceding claims wherein the heavy metal acetate/s in the sealing bath is/are lead, nickel and cobalt.

7. A process as claimed in any of the preceding claims hydric alcohol used in the sealing bath is mannitol or sorbitol.

Dated this 13th day of February 1975

R. Prabhakar Rao

PATENTS OFFICER,
COUNCIL OF SCIENTIFIC &
INDUSTRIAL RESEARCH

GIPPP/(C.P & D.C.I)/79-80/150

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No. OF SHEETS :- 1.
SHEET No :- 1.

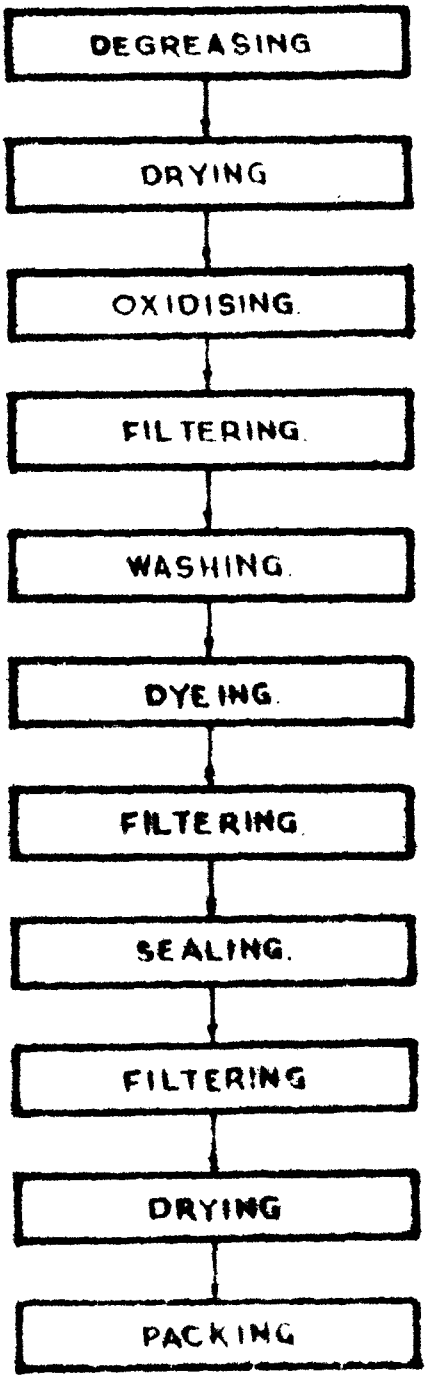
No 246/Cal/74

No. 141458.

FIG 1

FLOW SHEET

METHODS OF SEALING COLOURED ALUMINIUM POWDERS



(R.B. PAI)
PATENTS OFFICER,
C.S.I.R.