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### Provisional Specification

#### “IMPROVEMENTS IN OR RELATING TO COLOURING OF ALUMINIUM POWDER”

COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH, RAJ 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 BALKUNJE ANANTHA SHENOI, Scientist, and THIRUMANAMCHERI SESHADRI KRISHNAN, Senior Laboratory Assistant, both of the Central Electrochemical Research Institute, Karaikudi-3, India, both Indian citizens.

This invention relates to improvements in or relating to colouring of aluminium powder.

Hitherto it has been proposed to colour the aluminium powder in different ways:

(i) Colouring of aluminium powder can be done by mordanting it in a solution containing pyrogalllic acid or tannic acid. Later, the mordanted powder can be filtered and dyed using basic dyes like Rhodamine, malachite, green, auramine etc.

(ii) The mordanting can also be done with hot heteropoly acid derived from tungsten and later colouring it with basic dyes.

(iii) Bronze colours can be imparted to aluminium powder by first treating in chromic acid and then with potassium permanganate.

(iv) Colouring of aluminium powder can be done by using an isocyanate of molecular structure (RNCO) and both with water soluble and oil soluble dyes.

This is open to the objection that—

(i) in mordanting process using pyrogalllic and tannic acid as a mordanting agent and colouring with basic dyes, the basic dyes are to be in pure state and its cost is very high. Apart from this, the colour of the aluminium powder produced by adopting this process may not be stable, i.e. the colour will fade within a short duration.

(ii) in the process using hot aqueous solution of heteropoly acid derived from tungsten as a mordanting solution and then colouring with basic dyes, the phosphotungstic acid which is used as a mordant in this process is not available at lower cost. Apart from that, pure basic dyes are required.

(iii) in the chromic acid permanganate process, even though good bronze colour can be obtained, other colours like blue, red, green are not possible to get.

(iv) in the isocyanate process, the process involves chemicals like toluidine diisocyanate, 3,3', di-phenyl methane diisocyanate which are not available indigenously and hence have to be imported from foreign countries.

The object of the present invention is to obviate these disadvantages by using a simpler process for colouring of aluminium powder of different shades like gold, red, green, blue, violet etc. which involves abundantly available chemicals and which can be obtained from local market at a comparatively lower cost than those that are necessary for the processes hitherto mentioned.

To these ends, the invention broadly consists in oxidising of aluminium powder having different mesh sizes suitable for colouring. The oxidation is carried out in a bath which consists of the following chemicals: (i) methanol, (ii) water, (iii) sodium aluminate, (iv) sodium hydroxide, (v) sodium oxalate or sodium salicylate or sodium succinate.

The powder is degreased and stirred well in the above said bath for about 30 minutes to 180 minutes at 30°C to 65°C depending upon the mesh size and quantity of the powder used. The oxidised powder is filtered, washed thoroughly with tap water and then in distilled water until all the alkali is removed. Later, it is coloured with conventional dyes used for anodised aluminium. The colour and concentration of the dyes can be changed according to the requirements. Finally, the coloured powder can be washed, sealed and dried in an air-oven.

The following typical examples are given to illustrate the invention but not to limit the scope of the invention:

#### Example 1

120 grams of MS 7508 aluminium powder having -100 to +150 B.S. mesh size is degreased in acetone and dried at room temperature. The dried powder is stirred vigorously in one litre of the bath which is prepared with the following chemicals:

Methanol . . . . .	55% w/v
Sodium hydroxide . . . . .	2% w/v
Sodium aluminate . . . . .	1% w/v
Sodium salicylate . . . . .	1.5% w/v
Temperature . . . . .	60°C
Time . . . . .	180 minutes

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

Anodal Red SB . . . . .	10 g/l
Temperature . . . . .	60°C
pH . . . . .	5.5-6.5
Time . . . . .	5-10 minutes

Fine red coloured powder from the dye bath is filtered, washed, sealed and dried in the conventional method adopted for anodising and colouring of aluminium.

#### Example 2

100 grams of MS 7512 aluminium powder having -120 to +200 B.S. mesh size is degreased in acetone and dried at room temperature. The dried powder is stirred well in one litre of the bath which is prepared with following chemicals:

Price: TWO RUPEES.

Methanol . . . . .	60% w/v
Sodium hydroxide . . . . .	0.5% w/v
Sodium succinate . . . . .	1.5% w/v
Temperature . . . . .	45°C
Time . . . . .	120 minutes

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

Keviluminium golden yellow . . . . .	6 g/l
Temperature . . . . .	55°C
pH . . . . .	5-6
Time . . . . .	4 minutes

Beautiful golden coloured powder is filtered from the dye bath then it is washed, sealed and dried in the conventional method adopted for anodising and colouring of aluminium.

### Example 3

100 grams of coarse aluminium flakes are degreased in acetone and dried at room temperature. The dried powder is stirred vigorously in one litre of the bath which is prepared with the following chemicals:

Methanol . . . . .	30% w/v
Sodium hydroxide . . . . .	3% w/v
Sodium aluminate . . . . .	1.1% w/v
Sodium oxalate . . . . .	2% w/v
Temperature . . . . .	35°C
Time . . . . .	30 minutes

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

Keviluminium green GBL . . . . .	9 g/l
Temperature . . . . .	60°C
pH . . . . .	5.5-6.5

Fine green coloured powder from the dye bath is filtered, washed, sealed and dried in the conventional method adopted for anodising and colouring aluminium. Similarly many other colours obtained by anodising dyes can be imparted to aluminium powder by using this technique.

The following are among the main advantages of the invention:

1. The chemicals involved in this process are indigenously available.
2. This can be used as a substitute for bronze powder which is used by the printing industries for printing on textiles, paper and leather which will give good adhesion when mixed with suitable binder to the surface to be printed.
3. Different colours can be produced on aluminium powder by adopting this process.
4. This can be used even in manufacturing aluminium enamel paints with different colours.

Dated this 13th day of February, 1973.

(Sd./-)

(R. BHASKAR PAI)

PATENTS OFFICER,

Council of Scientific & Industrial Research.

## COMPLETE SPECIFICATION

### "IMPROVEMENTS IN OR RELATING TO COLOURING OF ALUMINIUM POWDER"

COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH, RAJI 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 BALKUNJE ANANTHA SHENOI, Scientist, THIRUMANAMCHERI SESHADRI KRISHNAN, Senior Laboratory Assistant, both are Indian Nationals and are employed in the Central Electrochemical Research Institute, Karaikudi-623006, Tamil Nadu, India.

This invention relates to improvements in or relating to colouring of aluminium powder.

Hitherto it has been proposed to colour the aluminium powder in different ways: (1) colouring of aluminium powder can be done by mordanting it in a solution containing pyrogalllic acid or tannic acid. Later, the mordanted powder can be filtered and dyed using basic dyes like Rhodamine, malachite green, auramine etc. (2) The mordanting can also be done with hot heteropoly acid derived from tungsten and later colouring it with basic dyes. (3) Bronze colours can be imparted to aluminium powder by first treating in chromic acid and then with potassium permanganate. (4) Colouring of aluminium powder can be done by using an isocyanate of molecular structure (RNCO) and both with water soluble and oil soluble dyes.

The main drawbacks connected with hitherto known processes are:

1. In mordanting process using pyrogalllic and tannic acid as a mordanting agent and colouring with basic dyes, the basic dyes are to be in pure state.

Apart from this, the colour of the aluminium powder produced by adopting this process is not stable, i.e. the colour will fade away within a short duration.

2. In the process using hot aqueous solution of heteropoly acid derived from tungsten as a mordanting solution and then colouring with basic dyes, the phosphotungstic acid which is used as a mordant in this process is not available at a low cost. Apart from that, the basic dyes are not available indigenously.

3. In the chromic acid-permanganate process even though good bronze colour can be obtained other colours like blue, red, green are not possible to get.

4. The isocyanate process involves chemicals like toluidine di-isocyanate, 3,3' diphenyl methane di-isocyanate which are not available indigenously and they have to be imported.

The main object of this invention is to obviate these disadvantages by using a simpler process for colouring of aluminium powder of different shades like gold, red, green, violet which involves abundantly available chemicals and which can be obtained from local market at a comparatively lower cost than that are necessary for the processes hitherto mentioned.

The main finding underlying the invention consists in oxidising of aluminium powder having different mesh sizes in a bath consisting of the following chemicals: (1) Methanol, (2) Water, (3) Sodium aluminate, (4) Sodium hydroxide and (5) Sodium oxalate or sodium salicylate or sodium succinate.

The new result from the new finding is that an oxide layer which has highly absorbent characteristics suitable for dyeing is produced on aluminium powder.

According to present invention, there is provided a process for colouring aluminium powders of different mesh size by oxidising the aluminium powder in a bath consisting of methanol, and any one or more of sodium aluminate, sodium hydroxide, sodium oxalate, sodium salicylate, sodium succinate, at a temperature of 35-60°C for 30 to 180 minutes and subsequently colouring the same in organic dyes followed by filtering, washing, sealing and drying.

The novel feature of this process is that the coloured powders can be used as a substitute for bronze powders which is used by the printing industries for printing textiles, paper and leather.

The flow-sheet of the process is given below:

Cleaning  
Oxidising  
Filtering  
Washing  
Dyeing  
Sealing  
Drying                      Packing

The following typical examples illustrate how the invention is carried out in actual practice:

#### Example 1

120g of MS 7508 aluminium powder having -100 to +150 BS size is degreased in acetone and dried at room temperature. The dried powder is stirred vigorously in one litre of the bath which is prepared with the following chemicals:

Methanol . . . . .	55% w/v
Sodium hydroxide . . . . .	2% w/v
Sodium aluminate . . . . .	1% w/v
Sodium salicylate . . . . .	1.5% w/v
Distilled water . . . . .	Remaining %
Temperature . . . . .	60°C
Time . . . . .	180 minutes

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:

Anodal Red 5B . . . . .	10 g/l
Temperature . . . . .	60°C
pH . . . . .	5.5 to 6.5
Time . . . . .	5-10 minutes

Fine red coloured powder from the dye bath is filtered, washed, sealed and dried in the conventional method adopted for anodising and colouring of aluminium.

#### Example 2

100 gms of MS 7512 aluminium powder having -120 to +200 BS mesh size is degreased in acetone and dried at room temperature. The dried powder is stirred well in one litre of the bath which is prepared with the following chemicals:

Methanol . . . . .	60% w/v
Sodium succinate . . . . .	0.5% w/v
Distilled water . . . . .	remaining%
Temperature . . . . .	45°C
Time . . . . .	120 minutes

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

Keviluminu golden yellow . . . . .	6 g/l
Temperature . . . . .	55°C
pH . . . . .	5-6
Time . . . . .	4 minutes

Beautiful golden coloured powder is filtered from the dye bath. Then it is washed, sealed and dried in the conventional method adopted for anodising and colouring of aluminium.

#### Example 3

100 gms of coarse flakes are degreased in acetone and dried at room temperature. The dried powder is stirred vigorously in one litre of the bath which is prepared with the following chemicals:

Methanol . . . . .	30% w/v
Sodium aluminate . . . . .	1.1% w/v
Sodium hydroxide . . . . .	3% w/v
Sodium oxalate . . . . .	2% w/v
Distilled water . . . . .	remaining %
Temperature . . . . .	35°C
Time . . . . .	30 minutes

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

Keviluminu green SBL . . . . .	9 g/l
Temperature . . . . .	60°C
pH . . . . .	5.5-6.5

Fine green coloured powder from the dye bath is filtered, washed, sealed and dried in the conventional method adopted for anodising and colouring aluminium.

The main advantages of the invention are:

1. The chemicals involved in this process are indigenously available.
2. This can be used as a substitute for bronze powder which is used by the printing industries for printing on textiles, paper and leather.
3. Different colours can be produced on aluminium powder by adopting this process.
4. This can be used even in manufacturing aluminium enamel paints with different colours.

This process consists of oxidising aluminium powder in a bath containing methanol, sodium aluminate, sodium hydroxide and sodium oxalate or salicylate or succinate and then filtering, washing, dyeing, sealing and drying.

#### WE CLAIM:

1. A process for colouring aluminium powder of different mesh size by oxidising the aluminium powder in a bath consisting of methanol, and one or more of sodium aluminate, sodium hydroxide, sodium oxalate, sodium salicylate, sodium succinate at a temperature of 35-60°C for 30-180 minutes and subsequently colouring of the same in organic dyes followed by filtering, washing, sealing and drying.
2. A process as claimed in claim 1 wherein the oxidising bath consists of methanol 30-60% w/v, sodium hydroxide 0.5-3% w/v, sodium aluminate 1.0-1.1% w/v, sodium succinate 1.5% w/v, sodium oxalate 2% w/v, sodium salicylate 1.5% and the remaining, distilled water.

Dated this 12th day of February, 1974.

(Sd./-)

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