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"IMPROVEMENTS IN OR RELATING TO PREPARATION OF  
INHIBITIVE PIGMENTS".

Council of Scientific and Industrial Research, Rafi Marg,  
New Delhi, 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 Kummattithidal Santhanam Rajagopalan  
Subbiah Nadar Guruviah, Meyyappa Sundaram, Scientists and Venka-  
tasubramanian Chandrasekaran, Senior Scientific Assistant - all  
from ~~Central~~ Central Electrochemical Research Institute, Karaikudi  
Tamil Nadu, India and Indian citizens.

PRICE: TWO RUPEES

143324

This invention relates to Improvements in or relating to Inhibitive Pigments.


Hitherto, it has been proposed to use red lead, litharge zinc chromate, barium chromate, calcium chromate, strontium chromate, calcium plumbate as inhibitive pigments which are costly.

The object of the present invention is to obviate this disadvantage by making an inhibitive pigment which is much less in cost than that of the inhibitive pigments mentioned above.

To these ends, the invention consist by taking one part of chrome ore ( $\text{Cr}_2\text{O}_3 = 50.68\%$ ,  $\text{FeO} = 14.37$ ,  $\text{SiO}_2 = 3.4\%$ ,  $\text{Al}_2\text{O}_3 = 10.86\%$ ,  $\text{MgO} = 13.5$ ,  $\text{CaO} = 1.06\%$ ) obtained from Ferro-Alloy Corporation, Srikakulam, Andhra Pradesh, ground and sieved to -300 mesh with 0.5-1.0 part of quick lime sieved to -300 mesh, mixing the two well in a ball mill for 4 hours and heating the mix in a furnace at  $750^\circ\text{C}$ - $900^\circ\text{C}$  in presence of air, with wetting by distilled water at the end of each hour and carrying out the reaction for 4 hours. The following typical examples are given to illustrate the invention.

Example 1:

100 gm., of -300 mesh sieved chrome ore obtained from Srikakulam and 37 gm., of -300 mesh sieved calcium oxide

were mixed well in a ball mill for 4 hours. This mixture was then placed inside a muffle furnace and the furnace was switched on. The mixture was heated in the furnace at  $850\text{C} \pm 20^{\circ}\text{C}$ . for 4 hours during which period, air from a compressor was blown into the furnace through a hole. At the end of each hour the furnace was opened, the tray containing the mixture was brought to the edge of the furnace and distilled water was sprayed over the mixture. After this the tray was placed in its original position viz., the centre of the furnace, and heating continued. The spraying procedure was repeated two more times during the 4 hour heating. The product was analysed for soluble chromate and alkalinity.  The chromate extracted is 500-600 mg/100 ml and the alkalinity is 0.015 N.

#### Example 2

100 gm., of -300 mesh sieved chrome ore obtained from Sriekulam were mixed with 28 gm., of -300 mesh sieved calcium oxide in a ball mill for 4 hours. The mixture was placed inside a muffle furnace kept at  $750 \pm 20^{\circ}\text{C}$ . for 4 hours during which period air from a compressor was blown into the furnace through a hole. At the end of each hour, the furnace was opened and distilled water was sprayed over the mixture after having brought the tray containing the mixture to the open end of the furnace. After spraying the tray was again placed inside the furnace in its original position and heating continued. The spraying procedure was repeated two more times during the four hour heating. The analysis of the aqueous extract for soluble chromate and alkalinity show that the chromate extracted is 400-500 mg/100 ml and alkalinity is 0.003N.

\* 30 gm., of the pigment prepared were shaken with 100 ml of distilled water for 6 hours and filtered. The filtrate was used for the analysis. The chromate was estimated by titrating against standard thiosulphate and alkali by titrating against standard acid.

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OBSERVATION AT THE END OF 30 daysImmersion Studies in

Painted Mild Steel Plates	Distilled Water	NaCl 3%	Salt Spray
1) Two coats of chrome ore paint in epoxy ester.	No blisters and no rust at scratch	No blisters <sup>†</sup> No rust at scratch	No change No blisters
2) Two coats of $\text{CaCrO}_4 \cdot \text{Fe}_2\text{O}_3$ primer in epoxy ester	Few fine blisters No rust at scratch	No blisters No rust at scratch	not affected

Advantages:

1. The inhibitive pigment developed is much less costly (Rs 4/kg) than all other known inhibitive pigments (Rs 10-Rs16/kg)
2. The primer prepared using this inhibitive pigment gives corrosion protection comparable to calcium chromate-iron oxide primer (Rs10.50/kg) which itself is the least costly among the other inhibitive primers (Refer table)

Dated this 26th day of March, 1975

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Asst. Patent Officer,  
Council of Scientific & Industrial Research

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THE PATENTS ACT 1970

COMPLETE SPECIFICATION

(See Section 10)

" IMPROVEMENTS IN OR RELATING TO PREPARATION OF  
INHIBITIVE PIGMENTS "

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 Kummattihidal Santhanam Rajagopalan,  
Subbiah Nadar Guruviah, Meyyappa Sundaram, Scientists and Venka-  
tasubramanian Ghandrasekaran , Senior Scientific Assistant- all  
from Central Electrochemical Research Insititute, Karaikudi  
Tamil Nadu, India and Indian citizens.

This invention relates to improvements in or relating to preparation of inhibitive pigments.

Hitherto, it has been proposed to use red lead, litharge, zinc chromate, barium chromate, calcium chromate, strontium chromate, calcium plumbate as inhibitive pigments which are costly. Some extenders like bentonite, soap stone iron oxide are added separately to the inhibitive pigment to reduce the cost.

The object of the present invention is to obviate the disadvantage by preparing the inhibitive pigment along with extenders in <sup>Si</sup>~~in~~ <sub>^</sub> situ in one operation to reduce the cost and improve the performance of the primer paint.

The main finding of the invention is the preparation of inhibitive pigment in situ by reacting chrome ore with a quick lime at high temperature in presence of air and with intermittent water spray.

This process eliminates the separate addition of extender to the pigment, since the low cost inhibitive pigment obtained by this process contains both the inhibitive component ( $\text{CaCrO}_4$ ) and the extender ( $\text{Fe}_2\text{O}_3$ ). In other words in situ blending of pigment and extender takes place during the preparation of the pigment by this process.

The invention broadly consists in mixing -300 mesh powder of one part of chrome ore ( $\text{Cr}_2\text{O}_3$  45-50%,  $\text{FeO}$  14-16%  $\text{SiO}_2$  3-6%,  $\text{Al}_2\text{O}_3$  4-12%,  $\text{MgO}$  12-14%,  $\text{CaO}$  1-2%) with 0.5-1.0 part of quick lime of -300 mesh in a ball mill for one hour and heating the mix in a furnace at <sup>de</sup> 750°C-900°C in presence of air, with wetting by <sup>de</sup> ~~the~~ <sub>^</sub> ionised water at the end of each hour and carrying out the reaction for 4 hours. The following typical examples are given to illustrate the invention. An inhibitive pigment (calcium chromate/Iron oxide) can be prepared in one step.

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Example 1

100 gm of -300 mesh sieved chrome ore and 37 gm of -300 mesh sieved calcium oxide were mixed well in a ball mill for one hour. This mixture was then placed inside a muffle furnace and the furnace was switched on. The mixture was heated in the furnace at  $850^{\circ}\text{C} \pm 20^{\circ}$  for 4 hours during which period, air from a compressor was blown into the furnace through a hole. At the end of each hour the furnace was opened, the tray containing the mixture was brought to the edge of the furnace and deionised water was sprayed over the mixture. After this the tray was placed in its original position viz., the centre of the furnace, and heating continued. The spraying procedure was repeated two more times during the 4 hour heating. The product was analysed for soluble chromate and alkalinity.\* The chromate extracted is 500-600 mg/100ml and the alkalinity is 0.015 N.

Example 2

100 gm of -300 mesh sieved chrome ore were mixed with 28 gm of -300 mesh sieved calcium oxide in a ball mill for 4 hours. The mixture was placed inside a muffle furnace kept at  $750 \pm 20^{\circ}\text{C}$  for one hour during which period air from a compressor was blown into the furnace through a hole. At the end of each hour, the furnace was opened and deionised water was sprayed over the mixture after having brought the tray containing the mixture to the open end of the furnace. After spraying the tray was again placed inside the furnace in its original position and heating continued. The spraying procedure was repeated two more times during the four hour heating.

\* 30 gm of the pigment prepared were shaken with 100 ml of distilled water for 6 hours and filtered. The filtrate was used for the analysis. The chromate was estimated by titrating against standard thiosulphate and alkali by titrating against standard acid.

The analysis of the aqueous extract for soluble chromate and alkalinity show that the chromate extracted is 400-500 <sup>mg</sup> /100ml and alkalinity is 0.003 N.

The calcium chromate/iron oxide pigment prepared by this method was tested for its requirement as pigment. The results are given in Table 1.

Table 1

S. No	Characteristic property	Values obtained	Method of Test IS 33-63
1	Volatile matter, percentage by weight, max.	0.35-0.40	6.1
2	Residue on sieve, percentage by weight, max (300 mesh)	0.5-1	7.2
3	Oil absorption	50-55	8.2
4	Colour	Chocolate	9.1
5	Water soluble sulphates chlorides (as $\text{SO}_4^{2-}$ , $\text{Cl}^-$ )	Nil	A.5
6	Calcium chromate solubility (as $\text{CrO}_3$ )	230-250mg when 30 gm of $\text{CrO}_3$ product was shaken with 100 ml of water for one hour.	A.3
7	Alkalinity	0.004-0.01	

The paint was prepared by incorporating this inhibitive pigment in epoxy ester medium, and its performance was evaluated by accelerated tests. This was compared with that of  $\text{CaCrO}_4/\text{Fe}_2\text{O}_3$  paint prepared by earlier method. (Indian patent No 118478) The performance of this paint at the end of 30 days is given in Table 2.



Table 2

OBSERVATION AT THE END OF 30 DAYS

Painted mild steel plates:	Immersion in Distilled water	NaCl 3%	Salt Spray
1. Two coats of chrome ore paint in Epoxy ester (0.75 to 1mil)	No blisters and no rust at scratch	No blisters No rust at scratch	No change No blisters
2. Two coats of $\text{CaCrO}_4$ / $\text{Fe}_2\text{O}_3$ primer in epoxy ester (0.75 to 1mil)	Few fine blisters. No rust at scratch	No blisters No rust at scratch	Not affected

The main advantages of the invention are:-

- 1) The chrome ores available in the country can be utilised to produce an inhibitive pigment which may be less costly than the conventional pigment.
- 2) The process produces both the inhibitive /inert pigments.

We claim :-

1. A process for preparing inhibitive pigment (for making primer paint which consists in mixing fine powder (-300mesh) of chrome ore such as herein described and quick lime in the ratio of 1:0.5-1.0 in ball mill and heating the resultant mixture in a furnace at  $750^{\circ}\text{C}$ - $900^{\circ}\text{C}$  in presence of air with periodical wetting with deionised water.
2. A process as claimed in claim 1 which consists in mixing fine powder of chrome ore (-300mesh) and quick lime in the ratio of 1:0.5-1.0 in a ball mill and heating the resultant material in a furnace at  $750^{\circ}\text{C}$ - $900^{\circ}\text{C}$  in presence of air from a compressor with periodical wetting by deionised water at the end of each hour and allowing the reaction to proceed for four hours, the final dried product is used for preparing paints.
3. A process as claimed in claim 1 wherein the ingredients are used in the following proportions at the specified temperature and duration:

Chrome ore  
Calcium oxide  
Temperature  
Duration

1 part  
0.75 part  
 $850^{\circ}\text{C}$   
4 hours

Dated this 5th day of April 1976

sd/-

Patents Officer