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(54) **Title**: SELF-CLEANING, ANTI-SMOG, ANTI-MOULD PHOTOCATALYTIC POWDERED WATER BASED PAINT

(57) **Abstract**: A photocatalytic powdered water based paint is described comprising photocatalytic binding cement, inert micronized limestone, low viscosity cellulose, fluidifying agent, anti-foaming agent, vinyl polymer and pigments. The water based paint is characterized by the fact of comprising at least one and preferably all the following further additives: metakaolin, titanium dioxide, calcium formate and diatomite.

TITLE

Self-cleaning, anti-smog, anti-mould photocatalytic powdered water based paint

DESCRIPTION

The present invention relates to a photocatalytic powdered water based paint that has considerable advantageous characteristics in addition to its main property of converting pollutants present in the air by photocatalysis, it being also able to remove bad smells, to prevent mould and bacterial cultures from developing on the supports painted therewith, as well as to prevent dark marks from developing on the surfaces because of its self-cleaning function and to prevent heat from being transmitted by reflecting the sunbeams.

Moreover the water based paint of the present invention is highly eco-friendly as it does not contain harmful products such as heavy metals (cadmium, mercury, arsenic, lead, hexavalent chromium), its content of volatile aromatic hydrocarbons (benzene and derivatives, toluene, o-xylene, styrene) is practically negligible, its content of volatile organic compounds (VOC) is less than 0,5 g/l, therefore it lends itself excellently to the application on any surfaces both made of stones and of metal or plastic, both for interiors and exteriors, in order not to alter over time the original appearance of buildings, monuments, public works of any kind.

Photocatalytic compositions have been known and used for some time for preserving the original appearance of cementitious, stone or marble structures protecting them from being attacked by pollutants in the air, by atmospheric agents and by the several difficulties generated by the present environmental conditions developed by the modern society. Examples of photocatalytic compositions based on titanium dioxide mixed with various additives are known for example in documents EP0784034, EP0633964, EP1196359, EP1944279, WO 2008/017934, WO 2009/080647 and WO 2010/012488. Although such compositions have led to satisfactory results, there is still the need for further improvements in order to overcome drawbacks, even important ones, that have not been solved by such compositions.

Among such drawbacks it is of particular importance the fact that their application is possible only by spraying them or by using a roller, while by the

use of the brush the product does not spread leaving the so called brush marks, that is brush strokes are in relief resulting in a not much satisfactory aesthetical appearance; moreover the maturing of the product and its mechanical strengths over time resulted in a chalking of the paint; other drawbacks were the difficulty in application, hiding power, non optimal point of white and mechanical performances; and the possibility of the formation of efflorescences due to the phenomenon of hydrolysis lime.

All these problems have been solved with a long lasting work studying problems, trials, tests and analyses, that have led to the selection of optimal ingredients and have led to the realization of the excellent photocatalytic water based paint of the present invention.

In order to achieve a real and effective improvement of the water based paint, several searches and tests have been accomplished on the several types of raw materials, starting from a standard laboratory mixture composed of photocatalytic cement as a binder and as fundamental matrix for photocatalysis development, micronized limestone and of very low viscosity cellulose, to which additives have been added from time to time in order to solve each problem initially related to such type of water based paint. Tests represented below show the real efficacy of the new additives in comparison to other additives of the same type that were used previously. The overall final mixture constituting the water based paint according to the present invention derives from all the new additives investigated in said tests that are incorporated therein and therefore, the composition of said final mixture, used later for performing the production tests, is shown below in this document, after discussing said tests.

In the following correlation table there is the list of all the ingredients used for performing the laboratory tests shown below:

Trade name	Type of product	Manufacturer
Cement TX	Photocatalytic binder with titanium dioxide	Italcementi SpA Bergamo, Italia
Micronized MIXER 100	Micronized limestone 20µ	Mineraria Ligure Srl Marina di Carrara, Italia
Melflux 2651 F	Polycarboxylic superfluidifying agent	BASF Construction Polymers Gmbh Trostberg Geramany
Titanium R-XL	Titanium dioxide	Tioxide Europe Srl Grosseto, Italia
Culminal mhpc 500 pf	Methylhydroxypropylcellulose	Hercules Aqualon division Bevern, Belgium
Tecnocell 500	Functional cellulose fibre	CFF Gmbh Gehren, Germany
Vinnapas 8034 H	Hydrophobic vinyl polymer	Wacker Chemie Italia Srl Milano, Italia
Calcium formate	Pure calcium formate	Dolder Massara Srl Varese, Italia
Celite 209	Diatomite	World Minerals Italia Srl Milano, Italia
Defoamex AP 199	Anti-foaming agent	Lamberti Spa Varese, Italia

Test 1 – Improvement in the hiding power of the product.

Test performed by application on a contrast card.

1. Weighing in two suitable containers the following components:

COMPOSITION A		COMPOSITION B	
Product	Weight	Product	Weight
Cement TX	1000 gr.	Cement TX	1000 gr.
Micronizzato C138M	1000 gr.	MIXER 100	1000 gr.
Culminal mhpc 500 pf	10 gr.	Culminal mhpc 500 pf	10 gr.
Melflux 2651F	6 gr.	Melflux 2651F	6 gr.
		Titanium R-XL	100 gr

2. Dry-mixing the components, naming them as follows: COMPOSITION A, COMPOSITION B.

Afterwards it will be possible to make the mix by means of laboratory mixing equipment (Hobart):

3. Placing 350 gr. of water in the Hobart and under agitation adding 1000 gr. of the sample called COMPOSITION A, after 15''verifying that there is no material adherent to the walls of the mixer, should this be the case removing it by a paddle and continuing the mixing at a high number of revolutions for further 120'' , settling for 60" and adding further 250 gr. of water with a low number of revolutions for 45".
4. Repeating step 3. with the sample called COMPOSITION B.
5. Placing a suitable amount of the mixed materials, on the contrast card making sure that they are adjacent one another and not overlapped one another.
6. By means of the 150 µ film applicator, spreading the material up to the end of the card.
7. After drying verifying that the material has covered the white to black contrast.

RAW MATERIALS	COMPOSITION A	COMPOSITION B
Cement TX	49.6%	47.25%
Micronized MIXER 100		47.25%
Micronized C138 M	49.6%	
Titanium R-XL		4.7 %
Culminal mhpc 500 pf	0.5 %	0.5%
Melflux 2651F	0.3 %	0.3 %

Tab. 1

Test results:

COMPOSITION A: contrast covered by 25%

COMPOSITION B: contrast covered by 75%

Final notes:

Table 1 schematically shows the compositions and their percentages by means of which the hiding power test has been performed. Composition B is definitely better because of the high point of white given by adding titanium dioxide, and because of the hiding power due to the micronized mixer 100 since it is finer than micronized C 138 M.

Test 2 – Improvement in the product fluidity.

Test performed by means of the Ford cup n.° 4

1. Weighing in a suitable container the following components:

Cement TX	1000 gr.
MIXER 100	1000 gr.
Culminal mhpc 500 pf	10 gr.

2. Dry-mixing the components and dividing them into two further samples each one of 1000 gr., naming them as follows: Cp. 1a, Cp. 2b.

3. Adding to the sample called Cp. 1a 4.0 gr. of MELMENT F 10, and dry-mixing it. Adding to the sample called Cp. 2b 3.0 gr. of MELFUX 2651F, and dry-mixing it.

Afterwards it will be possible to make the mix for performing the viscosity test by means of the Ford cup:

4. Placing 350 gr. of water in the Hobart (laboratory mixer) and

under agitation adding 1000 gr. of the sample called Cp. 1a, after 15''verifying that there is no material adherent to the walls of the mixer, should this be the case removing it by a paddle and continuing the mixing at a high number of revolutions for further 120'' , settling for 60" and adding further 250 gr. of water at a low number of revolutions for 45". With the obtained mix, reading the viscosity by Ford cup n. 4.

5. Repeating step 4. with the sample called Cp. 2b.

6. Writing the results and verifying which is the product with the highest fluidity.

RAW MATERIALS	Cp. 1a	Cp. 2b
Cement TX	49.65%	49.65%
Micronized MIXER 100	49.65%	49.65%
Culminal mhpc 500 pf	0.5%	0.5 %
Melment F 10	0.2%	
Melflux 2651F		0.2%

Tab. 2

Test results:

Viscosity after test by Ford cup Cp. 1a: 80"

Viscosity after test by Ford cup Cp. 2b: 40"

Final notes:

Table 2 schematically shows the compositions and their percentages by means of which the viscosity test by Ford cup n.4 has been performed; it can be clearly seen by the results that, although having a low dosage, the product containing MELFLUX 2651F (Cp. 2b) makes the system more fluid, therefore increasing the processability of the product and improving the final aesthetical appearance.

Test 3 – Improvement of the setting time and of the mechanical strengths of the product.

Test performed by means of Vicat needle.

1. Weighing in two suitable containers the following components:

COMPOSITION 1A		COMPOSITION 1F	
Cement TX	1000 gr.	Cement TX	1000 gr.
MIXER 100	1000 gr.	MIXER 100	1000 gr.
Culminal mhpc 500 pf	10 gr.	Culminal mhpc 500 pf	10 gr.
Melflux 2651F	3 gr.	Melflux 2651F	3 gr.
		Calcium formate	40 gr.

2. Dry-mixing the components naming the obtained products as COMPOSITION 1A and COMPOSITION 1F.

Afterwards it will be possible to make the mix in order to take the setting time by means of Vicat needle:

3. Noting down the time when the test begins, placing 350 gr. of water in the Hobart (laboratory mixer) and under agitation adding 1000 gr. of the sample called COMPOSITION 1A, after 15''verifying that there is no material adherent to the walls of the mixer, should this be the case removing it by a paddle and continuing the mixing at a high number of revolutions for further 120'' , settling for 60'' and adding further 250 gr. of water at a low number of revolutions for 45''.

4. Placing the obtained mix in the frustum of cone of the Vicat needle, that has been previously calibrated.

5. Bringing the plunger in contact with the mix and releasing the plunger such that it can penetrate into the mix, reading the penetration, noting down the time and penetration, cleaning the plunger.

6. Repeating step 5. at regular time periods decreasing the rest time between one penetration and the following one after setting has started. The setting is said to have started when the penetration of the plunger is not more at full scale, but it stops at least by 2 mm., the test is said to have ended when the plunger is not more able to penetrate into the mix by at least 3 mm.

Repeating steps 3., 4., 5., 6., with the sample called as COMPOSITION

1F.

In this case, due to the length of the test, an automatic Vicat needle has been used, by setting the penetration time, then data have been stored by the internal software.

RAW MATERIALS	COMPOSITION 1A	COMPOSITION 1F
Cement TX	49.68%	48.73 %
Micronized MIXER 100	49.68%	48.73 %
Culminal mhpc 500 pf	0.49%	0.49%
Melflux 2651F	0.15%	0.15 %
Calcium formate		1.90 %

Tab. 3

Test results:

	Start of setting	End of setting
COMPOSITION 1A	6 h 30′	12 h 20′
COMPOSITION 1F	5 h 30′	7 h 20′

Tab. 4

Final notes:

Table 3 schematically shows the compositions and their percentages by means of which the setting time test has been performed, and table 4 shows the results; from these results it can be clearly noted that the calcium formate plays an important reaction role with the cement paste causing the paste to set more quickly, such situation allows the final paint to be exposed earlier to the

atmospheric agents without being subjected to considerable changes as it occurs with long setting time such as the case of the composition 1A.

Test 4 – Improvement of the resistance to water.

Water drop test on the contrast card

1. Weighing in two suitable containers the following components:

COMPOSITION A		COMPOSITION B	
Cement TX	1000 gr.	Cement TX	1000 gr.
MIXER 100	1000 gr.	MIXER 100	1000 gr.
Culminal mhpc 500 pf	10 gr.	Culminal mhpc 500 pf	10 gr.
Melflux 2651F	6 gr.	Melflux 2651F	6 gr.
Pentaresin P3	100 gr.	Vinnapas 8034H	100 gr.

2. Dry-mixing the components, naming them as follows: COMPOSITION A, COMPOSITION B.

Afterwards it will be possible to make the mix by means of laboratory mixing equipment (Hobart):

3. Placing 350 gr. of water in the Hobart and under agitation adding 1000 gr. of the sample called COMPOSITION A, after 15''verifying that there is no material adherent to the walls of the mixer, should this be the case removing it by a paddle and continuing the mixing at a high number of revolutions for further 120'' , settling for 60'' and adding further 250 gr. of water at a low number of revolutions for 45''.
4. Repeating step 4. with the sample called COMPOSITION B.
5. Placing a suitable amount of the mixed materials, on the contrast card that has been previously named with the type of composition under testing.
6. By means of the 150 μ film applicator, spreading the material up to the end of the card.
7. After drying, pouring some water drops on the applied film, after its evaporation investigating the film condition.

RAW MATERIALS	COMPOSITION A	COMPOSITION B
Cement TX	47.25%	47.25%
Micronized MIXER 100	47.25%	47.25%
Culminal mhpc 500 pf	0.47%	0.47%
Melflux 2651F	0.3 %	0.3 %
Pentaresin P3	4.73%	
Vinnapas 8034H		4.73%

Tab. 5

Test results:

COMPOSITION A: Partial softening of the film after evaporation of the water drop.

COMPOSITION B: In some case a slight mark after evaporation of the water drop.

Final notes:

Table 5 schematically shows the compositions and their percentages by means of which the water drop test has been performed. The composition B yields a better performance the film does not undergo any softening and the mark generated only in cases with a larger drop.

By combining together the results obtained from tests 1-4, shown above, therefore it has been decided to add to the standard mixture the new additives tested in said tests and specifically the titanium dioxide and the fine micronized limestone in order to improve the product hiding power (Test 1), the polycarboxylic acid-based fluidifying agent in order to improve the product viscosity (Test 2), the calcium formate in order to improve the setting time and the mechanical strengths of the product (Test 3) and the hydrophobic vinyl polymer in order to improve the resistance to water of the product (Test 4).

Thus we arrived to the final formulation of the excellent water based paint of the present invention, summarized in the following table, wherein the

percentages by weight are the optimal and preferred ones but they are not limitative.

RAW MATERIALS OF THE INVENTIVE FORMULATION	From %	To %
Cement TX – photocatalytic binder	40	50
Micronized Mixer 100 – limestone more fine and with a higher hiding power – Test 1	45	55
Power Pozz White - metakaolin	1,5	10
Titanium dioxide – hiding white – Test 1	1,5	10
Culminal mhpc 500 pf – very low viscosity cellulose	0,05	1,5
Calcium formate – set accelerator – Test 3	0,5	5
Tartaric acid – set retarder	0,05	1,5
Melflux 2651F – polycarboxylic acid fluidifying agent – Test 2	0,1	1,2
Vinnapas 8034 – hydrophobic vinyl polymer – Test 4	3	10
Deofoam AP199 – anti-foaming agent	0,1	2,8
Tecnocell 500 – low viscosity cellulose fibres	0,1	1,2
Celite - diatomite	0,5	3,5
Immediately dispersible pigments Pantocrom	q.s.	q.s.

Thus we have come to the final formulation of the excellent water based paint of the present invention. As regards the production of the water based paint from the production perspective, the mixing plant of the applicant located in Marcellina, province of Rome, has been used; after having found the ideal composition in laboratory after the tests previously described, it has been necessary to store the raw materials to be used into the storage silos that have been accurately emptied and cleaned, then the recipe has been stored in the production managing software, (recipe code n.° 75) such that all the weighings of the recipe occur in a completely automated manner reducing to zero the possibilities of doing weighing errors, the production scales MEMOMATIC model type 7053 called as scale 1, scale 2, scale 3, as the mixer model M-TEC type MR 150V serial number 9161092002 are subjected to a annual calibration and inspection. The production process can be summarized as follows:

- Automatically weighing the cement by means of a charging feeder directly on the scale 1 ;
- Unloading the scale 1 into the mixer;
- Automatically weighing the additives by means of charging feeders directly on scale 3;
- Unloading the scale 3 into the mixer;
- Automatically weighing the micronized by means of a charging feeder directly on scale 2;
- Unloading the scale 2 into the mixer;
- Starting to mix firstly only by the help of the mixing blade, then even by means of three turbulators provided within the mixer (the blade serves for roughly mix the raw materials inserted therein, while the turbulators allow even the finest parts of the components to be mixed more closely)
- Unloading the mix into the storage silos.

The mixing time of the blade and of the turbulators have been set on the basis of the acquired experience in the following mode:

Blade mixing time 180"

Turbulator mixing time 180".

In order to verify the complete homogeneity of the components several material samples have been taken in various phases during the paint canning, and after having mixed the material using always the same method used in the laboratory tests, viscosity has been investigated, we have obtained the same value in each case, therefore the material is completely homogeneous in the whole production lot thereof.

Table 6 shows the values detected during the production control.

Tab. 6.

SAMPLE	VISCOSITY
START	55"
SAMPLE 1/3	55"
SAMPLE 2/3	55"
END	55"

Therefore it is possible to conclude that a very high quality photocatalytic water based paint has been obtained, helping in maintaining the air clean and the building faces more clean for a long time, decreasing their reconstruction costs, it is easy to be applied, it has considerably improved characteristics of hiding power, viscosity, water resistance, setting time and mechanical strength. It has to be pointed out also that the indication of the trade name of the several used additives is not to be considered as a limitation but as a mere example and such additives can be replaced by other products having like characteristics and meeting the same requirements set forth in the previous description and in the annexed claims.

CLAIMS

1. Photocatalytic powdered water based paint comprising photocatalytic binding cement, inert micronized limestone, low viscosity cellulose, fluidifying agent, anti-foaming agent, vinyl polymer and pigments, characterized in that it comprises at least one and preferably all the following further additives: metakaolin, titanium dioxide, calcium formate and diatomite.

2. Water based paint according to claim 1, wherein the ingredients are contained in the formulation in the percentages by weight shown in the following table

RAW MATERIALS OF THE INVENTIVE FORMULATION	From %	To %
Photocatalytic binding cement	40	50
Inert micronized limestone	50	55
Metakaolin	1,5	10
Titanium dioxide	1,5	10
Very low viscosity cellulose	0,05	1,5
Calcium formate	0,5	5
Tartaric acid	0,05	1,5
Polycarboxylic acid fluidifying agent	0,1	1,2
Hydrophobic vinyl polymer	3	10
Anti-foaming agent	0,1	2,8
Low viscosity cellulose	0,1	1,2
Diatomite	0,5	3,5
Immediately dispersible pigments	q.s.	q.s.

3. Water based paint according to the preceding claims, wherein the hiding power of the product is improved by adding titanium dioxide.

4. Water based paint according to the preceding claims, wherein the fluidity of the product is improved by adding polycarboxylic acid based fluidifying agent

5. Water based paint according to the preceding claims, wherein the setting time and the mechanical strength of the product are improved by adding calcium formate.

6. Water based paint according to the preceding claims, wherein the

water resistance of the product is improved by adding the hydrophobic vinyl polymer.

7. Water based paint according to the preceding claims, wherein the addition of pozzolanic metakaolin eliminates the formation of efflorescences.

8. Water based paint according to the preceding claims, wherein the addition of diatomite improves the homogeneity and opacity of the product.

9. Water based paint according to the preceding claims, wherein the use of pigments immediately dispersible when in contact with water prevents brush marks or coloured strips from being formed upon the application of the product.

10. Photocatalytic powdered water based paint according to the preceding claims, wherein after weighing individually successively automatically the cement, the additives and the micronized and after charging them into the mixer, the components are mixed firstly only by the help of the mixing blade for a first rough mixing of the ingredients and then by operating even the inner turbulators of the mixer in order to achieve a closer mixing even of the finest parts of the components, with equal mixing time in the two phases and by taking samples of the product in various phases during the canning process, the viscosity is verified to be constant thus proving the complete homogeneity of the product has been obtained.

INTERNATIONAL SEARCH REPORT

International application No
PCT/IT2011/000281

A. CLASSIFICATION OF SUBJECT MATTER
INV. C04B28/04
ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
C04B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)
EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 2009/013337 A2 (ITALCEMENTI SPA [IT]; ALFANI ROBERTA [IT]) 29 January 2009 (2009-01-29) pages 1-5 examples 1-9	1-10
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[X] Further documents are listed in the continuation of Box C.

[X] See patent family annex.

* Special categories of cited documents :

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Date of the actual completion of the international search	Date of mailing of the international search report
11 April 2012	24/04/2012

Name and mailing address of the ISA/	Authorized officer
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INTERNATIONAL SEARCH REPORT

International application No
PCT/IT2011/000281

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
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