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Characterisation of the variations of tint and the adhesion of pigments onto the surface of mortar

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HIGHLIGHTS

- Pigments transferred from the casting mould to the mortar are quantified by PIXE.
- Coupling PIXE and colorimetry helps in studying the variations of tint of mortar.
- A method based on water washing is proposed to check the adhesion of the pigments.

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ABSTRACT

A method, coupling Particle-Induced X-ray Emission (PIXE) and colorimetry, is proposed to determine the amount of pigments transferred from the casting mould and to quantify their adhesion onto the surface of coloured mortar about 4 weeks after demoulding. PIXE analyses allow the quantification of the amount of pigments anchored onto the hardened mortars, even if the observations by Scanning Electron Microscopy (SEM) remain necessary to study the near-surface microstructure, especially for the rough samples. The optical parameters deduced from the colorimetry measurements are more interesting to establish the homogeneity of the perceived tint. Indeed, a protocol is proposed to evaluate the adhesion of the pigments by relating the amount of pigments (detected by PIXE analyses) and the coordinates measured in the CIE (L^*, a^*, b^*) colour space, before and after a water washing test.

1. Introduction

With one m^3/habitant/year, concrete and mortar are the most widely used construction materials in the world, mainly because they offer the best compromise between production cost and performances. Although a few muralist precursors raised the question of mortar aesthetic at the beginning of the 20th century [1–4], it only became a major concern in the last decades [4–10]. The development of high-performance and self-compacting concretes [11–14], which show a significant improvement in terms of aesthetics, contributed to develop their use in architecture.

As soon as mortar starts to be a decorative material, the question of its colour becomes essential [6,10,15]. Two main ways are possible to colour the mortar: the colouring agent can be added in the volume (with a relative high cost as most of the material is wasted), or in the other hand, the pigments can be applied on the hardened mortar through a coating, which induces operational and environmental costs. Moreover, some types of pigments added in excessive amount can degrade the properties of the fresh concrete. According to the mix-design of mortar or concrete and the type of pigments, the ranges of the weight ratio between pigment and cement becomes acceptable between 4 and 9 wt.% [16,17]. This research considers new recent developments to decrease the amount of pigments used to colour the mortar or concrete. In this way, a new colouring process is developed, where pigments are dispersed during setting in the first micrometres of the surface only (<100 μm deep), with no painting, coating or varnishing step [4,18].

This study proposes to couple colorimetry measurements (already used to characterise the surfaces of mortar or concrete [15,19]) and ion beam analyses as PIXE (Particle-Induced X-ray Emission) to quantify the homogeneity of tint and the adhesion of pigments after hardening. The PIXE method, which allows the

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analysis of sample without performing any cross-section, is widely used to study cultural heritage artefacts [20] and has been used to characterise the composition of concrete during leaching [21]. The intrinsic properties of the pigments used and reference mortars (composition and colour) are described before characterising the surface of hardened mortars using colorimetry, Scanning Electron Microscopy (SEM) and PIXE analyses. Finally, colorimetry measurements and PIXE analyses were done before and after water washing in order to evaluate the adhesion of pigments onto the coloured mortars about 4 weeks after demoulding and to mimic the roles of rain and finger pressure.

2. Experimental section

2.1. Materials

2.1.1. Pigment

The choice of the pigments was based on several criteria. First, the atomic number of the elements of the pigment must be heavy to facilitate its characterisation from the microstructure of the mortar when observed in SEM by backscattered electrons (BSE). Moreover, the pigments must remain chemically inert to the mortar (which limits the choice to inorganic pigments) and must not fall in the area of heavy metals potentially dangerous for health (e.g. lead oxides). Indeed, a yellow-orange pigment, consisting of metal oxides composed of rutile TiO_2–ZnO–SnO_2 mixture [22], was chosen. Fig. 1a and b show the pigment under a camera and SEM: coarse grains with irregular shapes are visible.

2.1.2. Samples of mortar

A mix-design of mortar, based on white ultra-high performance concrete (UHPC), was selected by the fact that the surface properties – low roughness, dense particles packing with low porosity, white colour – should facilitate the characterisation of the samples [11,13]. The UHPC contains no coarse aggregates as standard mortars, but metal or organic fibres, which do not affect the surface properties; these fibres have been removed from the mix for laboratory tests. The mix-design of mortar was then made of 31 wt.% white Portland cement (CEM I 52.5 PMES), 9 wt.% of limestone filler, 7 wt.% of silica fume, 43.5 wt.% of sand (0–1 mm) and 1.5 wt.% of polycarboxylate-based admixture . A water to cement (W/C) weight ratio of 0.26 was used.

2.1.3. Casting parameters and demoulding solutions

Table 1 details different parameters (types of mould, compositions of the demoulding solutions, types of surfactants, and amounts of pigments) tested to manufacture the mortars and to establish the relationship between colorimetry

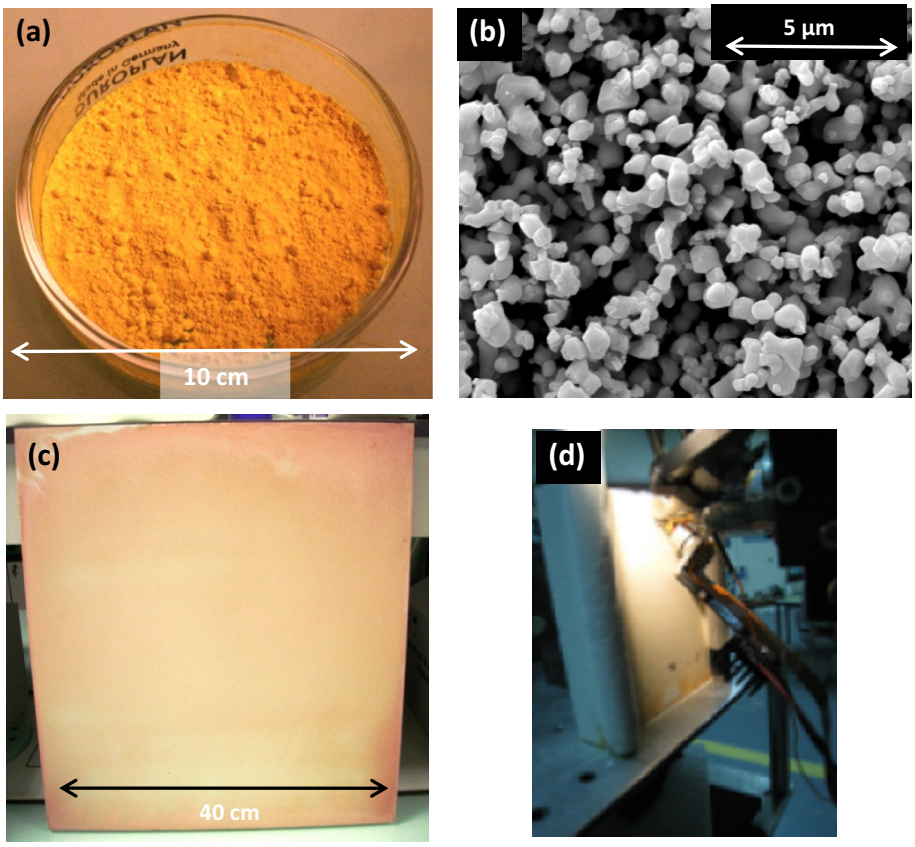

Fig. 1. (a) Images of: the pigment using camera; (b) using SEM in Secondary Electron mode; (c) the surface of a coloured mortar using camera; (d) a sample of mortar submitted to ion beam microanalysis using the particle accelerator AGLAE (C2RMF, Paris).

Table 1
List of the different casting parameters tested (types of mould, compositions of the demoulding solutions, types of surfactants, and amounts of pigments). R1 = linear hydrocarbon chain with 13 atoms of carbon; R2 = aliphatic hydrocarbon chain with 17 atoms of carbon; R3 = linear hydrocarbon chain with 12 atoms of carbon.

Casting mould	Compositions of the demoulding solutions, oil and surfactants	wt.% of surfactants compared to water	wt.% of pigments compared to demoulding solutions
Steel	Oil (pentaerithritol tetra oleate, terpineol and oleic acid)	/	0; 1; 5; 10; 20; 30
PVC	Water with surfactants A (R1–(O–CH_2–CH_2)$_3$–OH)	1; 5; 10; 25	
	Water with surfactants B (R1–(O–CH_2–CH_2)$_5$–OH)		
	Water with surfactants C (R1–(O–CH_2–CH_2)$_{10}$–OH)		
	Water with surfactants D (R2–(O–CH_2–CH_2)$_3$–OH)		
	Water with surfactants E (R3–N–(O–CH_2–CH_2–OH)$_2$)		