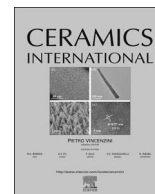




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## On a solar reflective ceramic based glaze for asphalt shingle

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### ABSTRACT

Solar reflective materials are one of the most effective solutions to counteract Urban Heat Island effect. Among them, asphalt shingles are one of the most widely used products. To improve solar reflectance of these surfaces usually both polymeric paint on the final product or ceramic glazes applied directly on the granules surface through rotary kiln are used. In this study the Design of Experiment approach is applied to an industrial formulation for ceramic glaze for asphalt shingles in order to find the optimal combination between pigment (Rutile and Talc), liquid phase (Sodium Silicate and Water) and heat treatment (700 °C –1100 °C). On the most significant samples, moreover, XRD and ESEM characterization has been performed in order to better understand the behaviour of the studied system. Interesting values in Solar reflectance were obtained, reaching  $\rho_{sol}=0.882$  creating a good solar reflective product ready to be applied, through rotary kiln, on mineral granules for asphalt shingles.

### 1. Introduction

The understanding and counteracting climate change is one of the big challenge of this century [1–5], not only in hot climates but also in cold climates [6,7]. In particular, the attention is focused on mitigation of Urban Heat Island effect [8,9], according to which temperature, in urban areas, is sensibly higher than in the surrounding rural areas. Several solutions were proposed to increase albedo and then trying to solve this problem [9]; among these the increase of green surfaces through “green roofs” and “green façades” can be found [10–15] which request together with higher setup costs a higher maintenance, or solar reflective surfaces or “cool surfaces” [16–18]. These are characterized by high solar reflectance and high thermal emissivity which allow to reject solar radiation back to the sky. Several roofing products can be modified to increase their solar reflectance [19,20] in order to match those requirements that can transform them in solar reflective materials. Among them, previous works improved the coatings for clay roof tiles and ceramic tiles both white and colored [21,22], formulating coatings with enhanced capability to reflect solar radiation across the whole solar spectrum (300–2500 nm). Asphalt shingle is another category of roofing products which can be easily turned into higher reflectance products [23–26]. To achieve this objective two ways can be pursued: the first consists on painting the final product with a polymeric high solar reflective coating, likely to what is widely applied on slow slope roof [27]. This solution, even quicker, presents some weakness since as every polymeric paint, the durability is pretty low

due to the limited resistance to UV radiation, moreover all the falling granules leaves black non reflective spots on the asphalt shingle surface. The second way is represented by the use of coated granules: a ceramic based solar reflective coating is applied through rotary kiln directly on the mineral granules which, once coated, are applied directly on the bitumen layer. This last strategy, although a little bit more complicated, guarantee a more effective and durable coating for the granules.

Using a ceramic coating is even one of the possible solutions to aging issues [28]: solar reflective surfaces, since exposed 24/7 h/day under the sun, are affected by aging processes which modify their exterior appearance through soiling layers and biological growth reducing the capability to reflect solar radiation.

Industrially, Titanium Dioxide is one of the most widely solar reflective pigment used [29]. Due to the high cost of this raw material a suitable replacement showing equal or higher reflecting properties becomes an urgent issue in this technological field, therefore the design of new industrial formulations based on an ceramic solar reflective coating applied onto basalt grit were explored in this paper. In particular, statistical based method such as Design of Experiment was used to optimize the solar reflectance of the final material as a function of the percentages of different components in the glass ceramic coating.

Multivariate statistical methods were recently introduced in materials science and technology to promote the rational explanation of the experimental results extracting the maximum amount of information

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from a complex data set. Among all these methods the Design of Experiments (DoE) covers an extremely interesting role allowing the obtainment of the maximum information performing a minimum set of experiments. In this perspective; DoE represents a real add value in materials design working as a complimentary tool to traditional approach often based on one factor variation a time (OFAT). [14].

Moreover, the Response Surface Methodology (RSM) inherent the method uses statistical and mathematical techniques to develop, improve and optimize processes associated often to the materials production. The correlations between the response property and the main variables (compositions, process parameters etc.) affecting the final performances of a selected material were described by complex mathematical function which, if validated, can play a predictive role [30–33].

In this work talc was selected as alternative to Titanium Dioxide due to its lower cost and its versatility. A pigment's partial replacement was studied together with the variation of liquid phase (water and Sodium Silicate) and heating treatment (three different isotherm temperatures) with the aim to obtain a product reaching the threshold of  $\rho_{\text{sol}}=0.882$  decreasing the cost impact in material production. The aim of this work is to quantitatively correlate the input variables to the final performances of the cool roof glaze in terms of solar reflectance to define the best process condition favoring the higher reflectance properties. The results clearly highlight the benefit to partially substitute talc to titania in term of potential application in cool roof formulation saving cost in raw materials.

## 2. Materials and methods

Traditionally, ceramic based coatings for asphalt shingles are applied through a rotary kiln where both mineral granulate and liquid glaze are heated letting the coating completely and homogeneously covers each single grain. To facilitate this process, usually, together with pigments, which in this case are represented by  $\text{TiO}_2$ , one of the most widely used white solar reflective pigment, kaolin, which help the glaze rheology, and water, liquid sodium or potassium silicate are added.

To obtain the samples according to the experimental plan reported in Table 1 the raw materials were poured in glass beker and magnetically stirred for 5 min, then the liquid glaze was dried at 110 °C, milled and sieved with a 64  $\mu\text{m}$  mesh sieve. Subsequently the powders were humidified at 6 wt% and compacted by uniaxial pressing to obtain circular (diameter 40 mm, thickness 0.5 mm) test samples. These

**Table 1**  
Experimental Plan.

N	Talc (wt%)	Water (wt%)	Temperature (°C)
1	15	35	1100
2	0	20	700
3	15	35	900
4	30	35	900
5	15	35	900
6	0	35	900
7	15	35	900
8	0	20	1100
9	15	35	700
10	15	50	900
11	15	20	900
12	15	35	900
13	15	35	900
14	15	35	900
15	0	50	1100
16	30	50	1100
17	0	50	700
18	30	20	1100
19	30	50	700
20	30	20	700

samples were heated at the temperature reported by the experimental plan in a cold to cold cycle with 10 °C/min heating ramp and a 15 min isotherm. Solar reflectance was measured on the disks, in accomplishment to ASTM E903 [35–37] with a UV–vis–NIR Spectrophotometer (Jasco V-670) equipped with a 150 nm integrating sphere. The solar reflectance was calculated (1) by integrating spectral reflectivity  $\rho_\lambda$  over the range from 250 to 2500 nm with a step size of 5 nm weighted by the spectral irradiance of the sun at the earth surface  $I_{\text{sol},\lambda}$  [ $\text{W}/(\text{m}^2 \times \text{nm})$ ] described by the AM1GH irradiance spectrum [36–38].

$$\rho_{\text{sol}} = \frac{\int_{300}^{2500} \rho_\lambda I_{\text{sol},\lambda} d\lambda}{\int_{300}^{2500} I_{\text{sol},\lambda} d\lambda} \quad (1)$$

Thermal behaviour of the system has been analyzed by Hot-Stage Microscope (HSM) (Misura, Expert System Solutions). For HSM analysis, powdered samples have been compacted to obtain small cylinders (3 mm of height and 1 mm of diameter). Subsequently they were heat treated from 20 °C to 1600 °C with a heating rate of 20 °C/min.

Microstructural characterizations were performed by means of Environmental Scanning Electron Microscope (FEI Quanta-200). The surface of the samples was analyzed after being sputtered with a 10 nm thick gold layer. Chemical analysis was carried out on the samples through X-EDS Oxford INCA-350.

Qualitative mineralogical analysis was conducted using a X'Pert Pro Panalytical diffractometer equipped with X'Celerator detector. CuK $\alpha$  radiation limited to 40 kV and 40 mA was used. The spectra were collected with a scanning rate of 0.1° min<sup>-1</sup> in a 2 $\theta$  range from 10° up to 80°.

## 3. Experimental design

Previous work reports a screening study which [34] point out the main role played by composition and its interaction on the solar reflectance of the glaze. For these reasons, pigment ratio (Titanium Dioxide and Talc), liquid phase ratio (Water and Sodium Silicate) and firing temperature (from 700 to 1100 °C) were considered as input parameters for RSM analysis. Details on the ranges considered in this study are reported in Table 2.

A central composite face-centered (CFC) design was considered to investigate data correlations and a quadratic model (1) was used as response surface in which  $\beta_0$  is the overall mean response,  $\beta_i$  is the main effect for each factor ( $i = 1, 2, \dots, p$ ),  $\beta_{ij}$  is the interaction between the  $i$ th and  $j$ th factors and  $\beta_{ii}$  is the quadratic effect for the  $i$ th factor.

$$Y = \beta_0 + \sum \beta_i x_i + \sum \beta_{ij} x_{ij} + \sum \beta_{ii} x_i^2 \quad (2)$$

The three different variables selected in this work as input factors were varied according to the Table 2. The experimental plan (Table 1) comprises 20 experiments including a center point replicated 5 times for the data reproducibility evaluation.

Talc has been introduced in substitution of  $\text{TiO}_2$  in formulations from 0 to 30 wt(%) according to previous statements deriving by screening analysis. [34]. On the other hand, the maximum and minimum values of water correspond to a minimum and maximum value of Sodium Silicate that is generally used in cool roof formulation. With respect to glaze composition, just two parameters (talc and water amount) were varied in the DoE experimental plan since the (wt%) of

**Table 2**  
Independent variables and limit level for response surface study.

Factor	Name	Type	Low Actual	High Actual
A	Talc	Numeric	0	30
B	Water	Numeric	20	50
C	Temperature	Numeric	700	1100

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