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A procedure for characterizing the curing process of cold recycled bitumen emulsion mixtures

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highlights and the second second

- A quantitative approach for measuring curing of cold recycled mixtures is proposed.
- Water and voids content had a limited impact on the evolution of indirect tensile strength.
- A good correlation was found between indirect tensile strength and water loss.
- The water evaporation process was virtually completed after 28 days.

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ABSTRACT

Due to the presence of water, emulsion and cement, cold recycled mixtures (CRM) are evolutive materials and thus require a certain curing time to develop their long-term properties. The present study describes a laboratory approach for characterizing the properties of CRM, focusing on their evolutive behaviour. The experimental activities were carried out in parallel at the École de technologie supérieure (ÉTS) in Montréal, Canada and at the Università Politecnica delle Marche in Ancona (UNIVPM), Italy. The evolution of water loss by evaporation and indirect tensile strength (ITS) was measured and analyzed using the Michalis-Menten model, in order to achieve a quantitative characterization of the curing process. The results showed that different dosages of water resulted in different rates of water loss by evaporation, but did not penalize the development of ITS. Moreover, for both CRM, a good correlation was found between water loss and ITS. Finally, the data showed that after 28 days of curing in the selected laboratory conditions, the evaporation process was virtually completed.

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1. Introduction

The term cold recycling is associated to a broad series of costeffective and sustainable technologies for the rehabilitation of bituminous pavements that are based on the use of reclaimed asphalt (RA) as main aggregate source and bitumen emulsion or foamed bitumen as main binders [\[1–3\].](#page--1-0) Ordinary Portland cement,

⇑ Corresponding author. E-mail address: a.graziani@univpm.it (A. Graziani). or other mineral additions like hydrated lime or fly ash, are generally used as secondary binders or as active fillers, in order to improve the mechanical properties of cold recycled mixtures (CRM). In the short term, the secondary binders allow an earlier opening to commercial traffic whereas in the long term they improve stiffness and strength of CRM layers [\[4–7\]](#page--1-0). Finally, water is an essential component of CRM, because during mixing it contributes to the homogeneous distribution of bitumen droplets within the mixture, and thus leads to a good coating of aggregates after emulsion breaking and setting $[8-10]$.

Because of the broad variability of component materials, mixture compositions and production techniques, the mechanical properties of CRM may be extremely variable. At least three mixture families can be identified: bitumen stabilized materials (characterized by high stress-dependency and failure in shear) [\[11\],](#page--1-0) cement-bitumen treated materials (stiffness and strength properties depend on the balance between bitumen and cement) [12-15] and cold-mix asphalts (aggregate fully coated with bitumen and high thermal sensitivity) [\[16,17\]](#page--1-0).

Despite such variability, a common distinctive feature of CRM is their evolutive behavior [\[18–22\].](#page--1-0) In fact, physical and mechanical properties of CRM require a certain curing time to evolve from the short-term "fresh" state, to the long-term "hardened" state. During curing, the water content reduces until reaching equilibrium with the surrounding environment within the pavement structure, whereas mechanical properties improve until attaining ultimate performance. Since both bituminous and hydraulic binders are generally present, the curing process of CRM occurs as a result of the interaction of different physical and chemical mechanisms, like emulsion breaking and setting and cement hydration.

Field curing is influenced by many factors, such as layer thickness, drainage condition, construction sequence, temperature and humidity. Thus, defining a curing process for laboratory investigations aimed at simulating field curing is very difficult. However, it is imperative to develop laboratory curing methods that allow to quantify differences in CRM behavior. This is obtained by fixing boundary conditions in terms of temperature and humidity, and carefully considering the effect of specimen-related properties like shape, dimensions and voids.

Bearing in mind the previous aspects, the curing process of CRM in the laboratory can be characterized in a quantitative way by identifying two parameters [\[23\]](#page--1-0):

- one parameter related to the CRM properties in the hardened state, i.e. at the end of the curing process;
- one parameter related to the initial rate of evolution of these properties, i.e. the initial rate of the curing process.

A similar approach is used to identify the evolution of Portland cement strength. For example, in the European Standard EN 197-1, one parameter (the compressive strength of a normalized mortar after 28 days of curing) is used to measure the strength in the hardened state and one attribute (N or R) is used to indicate the rate of strength increase at the beginning of the curing process.

1.1. Volumetric properties of CRM and their evolution

Volumetric properties of bituminous mixtures, either hot or cold, have a significant influence on their mechanical performance and durability. For cold mixtures, including CRM, it has to be considered that the volumetric composition changes during curing, mainly due to water evaporation [\(Fig. 1](#page--1-0)). In addition, part of the water that remains in the mixture actually changes its physical state because is used for hydration of Portland cement. In general, it is well known that the volume of the reaction products formed during hydration is slightly lower than the cement and water reacted [\[24\].](#page--1-0)

The solid phase of CRM includes RA (aged bitumen is considered part of the RA particles), virgin aggregate, filler and cement (or other secondary binders). The bitumen droplets in the emulsion, are actually ''solid" and is assumed that they are not absorbed by the aggregate which is wet condition at the mixing time. The total water in the CRM is the sum of the water from the bitumen emulsion and the water added during mixing. Part of the water is absorbed by the aggregate, the rest is intergranular water (also indicated as ''free" water), and is available to enhance workability, compaction and for cement hydration.

Curing causes a progressive increase of the volume occupied by the voids and the solid phase (products of cement hydration), whereas the volume of water progressively decreases, due to evaporation and cement hydration. Evaporation also causes a mass loss of the mixture that can be measured in the laboratory and in the field in order to monitor the curing process [\[25\].](#page--1-0) [Fig. 1](#page--1-0) shows typical changes of the volumetric composition of CRM over time.

The volumetric properties of CRM can be characterized by the voids in the mixture (V_m) and the voids filled with liquids (VFL), that are calculated as follows [\[9\]](#page--1-0):

$$
V_m = \frac{V_{V,A} + V_{W,F}}{V} = \frac{V - (V_S + V_{B,E})}{V}
$$
(1)

$$
VFL = \frac{V_{B,E} + V_{W,F}}{V_{V,A} + V_{B,E} + V_{W,F}} = \frac{V_{B,E} + V_{W,F}}{V - V_S}
$$
(2)

where V is the total volume of the specimen during compaction (geometric volume based on the height of the specimen calculated during compaction), V_S is the bulk volume of solids (aggregates and cement), V_{BE} is the volume of fresh bitumen (emulsion residue), V_{WF} is the volume of free water and V_{VA} is the volume of intergranular voids filled with air. It is highlighted that V_s , V_{BE} and V_{WF} are calculated using the initial mass of the materials and their density (or specific gravity), whereas V_{VA} is calculated based on the total volume of the specimen V (Eq. (2)). Therefore, the accuracy of the calculations carried out using Eqs. (1) and (2) relies on the hypothesis that material loss during compaction is negligible. This hypothesis needs to be carefully checked after the compaction of each specimen by comparing the mass of the material put in the mold and the mass of the compacted specimens.

1.2. Analytical model for curing description

In order to describe the curing process of CRM in a quantitative way, the development of physical and mechanical properties was analyzed using the Michaelis-Menten (MM) model [\[26\]](#page--1-0). The MM model was applied to describe the asymptotic increase of material properties, for example water loss by evaporation (WL) and indirect tensile strength (ITS), as a function of curing time t [\[23\]](#page--1-0) as follows:

$$
y(t) = \frac{y_A \cdot t}{h_y + t} \tag{3}
$$

where $y(t)$ is the material property under investigation, y_A is its asymptotic value and h_y is the curing time required for $y(t)$ to reach one half of the long-term value. The parameter h_v can be used as a measure of the initial curing rate: a lower value of h_v indicates a faster increase of y in the first curing stage, i.e. up to $y_A/2$. Both y_A and h_v have a clear physical meaning, hence their values must to be positive.

Eq. (3) may also be used to represent the relation between two material properties, for example ITS (dependent variable) as a function of WL (predictor). However, in this case, there is not an asymptotic increase and thus the regression parameters y_A and h_v do not have a clear physical meaning. In the initial curing stage, if ITS increases faster than WL, i.e. if $h_{\text{ITS}} < h_{\text{WL}}$, then Eq. (3) describes a curve which is convex upward (concave curve) [\[27\].](#page--1-0) This indicates that cement hydration (which is not directly related to water loss) has a marked effect on ITS increase rate. Vice versa, if $h_{WL} < h_{TIS}$, Eq. (3) describes a curve which is convex downward (convex curve). This indicates that emulsion breaking (which is directly related to water loss) controls the ITS increase rate. It can also be observed that, in the limiting case of $h_{\text{ITS}} = h_{\text{WL}}$,

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