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Method of quantification of hydrated lime in asphalt mixtures

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HIGHLIGHTS

- Only two methods to quantify hydrated lime in an asphalt mixture currently exist.
- The German method (acid–base titration of the extracted filler) was selected.
- The method was evaluated in a European round-robin test.
- Repeatability and reproducibility were 0.7% and 4.5% in absolute terms.
- The test method is robust and easy to implement.

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ABSTRACT

Hydrated lime has been known as an additive for asphalt mixtures for a long time and is now considered as an additive that increases asphalt mixture durability. It has been extensively used in the past 40 years in the USA, and is being increasingly used in most European countries, in particular Austria, France, the Netherlands, the United Kingdom and Switzerland. Given this context, it is necessary to have a fast and reliable quantification method of the hydrated lime content in an asphalt mixture.

A German method was used in order to do so. The test method consisted first in recovering the filler from the asphalt mixture using the usual solvent extraction method (EN 12697-1). Then, 1 g of the recovered filler was titrated with a 0.5 M HCl solution using a method adapted from EN 459-2. The test method was validated on an AC 10 mixture manufactured in the laboratory. The nominal content was 2.0% hydrated lime based on the dry aggregate. The measured content was found to be 1.7%, in reasonable agreement with expected results.

As a result, the hydrated lime content in an asphalt mixture can be evaluated. An estimate of the precision of the method is also given thanks to an international round robin test, showing that the repeatability of the method is close to 0.7% and its reproducibility 4.5% in terms of $Ca(OH)_2$ content in the recovered filler.

1. Introduction

Hydrated lime has been known as an additive for asphalt mixtures from their very beginning [1–3]. It experienced a strong interest during the 1970s in the USA, partly as a consequence of a general decrease in bitumen quality due to the petroleum crisis of 1973, when moisture damage and frost became some of the most pressing pavement failure modes of the time. Hydrated lime was observed to be the most effective additive [4] and as a consequence, it is now specified in many States and it is estimated that 40 Mt of asphalt mixtures are now produced in the USA each year with hydrated lime [5].

Given its extensive use in the past 40 years in the USA, hydrated lime has been seen to be more than a moisture damage additive [3,6–9]. Hydrated lime is known to reduce chemical ageing of the bitumen [3,6–8]. Furthermore, it stiffens the mastic more than normal mineral filler [3,6–8], an effect only observed above room temperature [3], that impacts the mechanical properties of the asphalt mixture.

Given that all the above mixture properties impact the durability of asphalt mixtures, the use of hydrated lime has a strong influence on asphalt mixtures durability [10]:

- North American State agencies estimate that hydrated lime at 1–1.5% in the mixture increases the durability of asphalt mixtures by 2–10 years, that is by 20–50% [5],

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- The French Northern motorway company, Sanef, currently specifies hydrated lime in the wearing courses of its network, because they observed that hydrated lime modified asphalt mixture have a 20–25% longer durability [11],
- Similar observations led the Netherlands to specify hydrated lime in porous asphalt [12], a type of mix that now covers 70% of the highways in the country.

As a result, hydrated lime is being increasingly used in asphalt mixtures in most European countries, in particular Austria, France, the Netherlands, the United Kingdom and Switzerland. The mechanisms behind this improvement in durability have recently been reviewed [13].

In this context, it is somewhat surprising that the problem of the quantification of hydrated lime in asphalt mixtures has only attracted interest in the recent years. As detailed below, only two methods can be found in the literature, both published less than 10 years ago: the first one comes from the USA and the second one, from Germany. In this work, we chose to use the German method in order to quantify hydrated lime in an asphalt mixture, because the testing equipment needed to put it into practice is usually already found in most road laboratories. In addition, its cost is very limited and the method could therefore be easily made available to a large number of control laboratories.

Therefore, this article first describes the published methods to quantify hydrated lime in an asphalt mixture. Then, it details the German method which has been first validated and then tested for repeatability and reproducibility in a new European round robin test.

2. Background: available methods to quantify hydrated lime in Asphalt Mixtures

2.1. US Method

The US method was developed by the Federal HighWay Administration (FHWA – [14,15]). It consists in measuring the Fourier Transform Infra-Red (FTIR) spectrum of the filler and quantifying the hydrated lime content from the peak intensity at 3,640 cm^{-1} corresponding to calcium hydroxide (Fig. 1). Calcium carbonate peaks at 1,390 cm^{-1} and can be unmistakably separated from hydrated lime (Fig. 1).

The analysis was shown to be easily performed by using 15–20 g of dust recovered by hammer drilling through an asphalt mixture with a 9.5 mm tungsten carbide bit [14,15].

Interestingly, measurements on 10 years old materials from Nevada showed that hydrated lime could still be detected after several years of traffic and weather exposure [14].

2.2. German method

As explained in more details below, the German method [16,17] is very simple and derives from the lime characterization methods detailed in EN 459-2 [18]. In fact, the German method separates three different characterization sub-methods:

1. Hydrated lime purity.
2. Hydrated lime content in a mixed filler.
3. Hydrated lime content in the filler recovered from an asphalt mixture.

The test consists in a hydrochloric acid titration of a suspension of the product to be tested. The acid has to be diluted (0.5 M) when mixed or recovered fillers are concerned, in order to adapt for a lower basicity. The filler is recovered from an asphalt mixture using solvent extraction of the bitumen as described in EN 12697-1 (usually using trichloroethylene or tetrachloroethylene as a solvent – [19]). The suspension to be titrated is then obtained by blending 1 g of recovered filler to 150 ml of water, 10 ml isobutanol and 5 ml of a surfactant solution (1 g sodium dodecylsulfate and 1 g polyethyleneglycol dodecylether in 100 ml water). The surfactant solution is needed only when recovered filler is tested, in order to wash out the filler from remaining bitumen or solvent from bitumen extraction. The colored indicator is phenolphthalein (0.5 g in 50 ml ethanol, completed to 100 ml by water). Titration rate is 12 ml/min initially, but decreases to 4 ml/min near the equivalence point. The method was shown to work with all types of fillers, including limestone filler [16].

A first national round robin test was performed in Germany with 12 laboratories [16]. The repeatability (in terms of absolute % weight of hydrated lime in the filler) was 0.52% and the reproducibility was 0.91% for a mean value of 27.3 wt.%.

The method was validated on samples taken out of cores 1.5 years after construction (Table 1 – [16]). The SMA 0/8 S mixes were made either with normal filler or with mixed filler containing 25% hydrated lime and the results are given in Table 1 [16].

Note also that a study using different methods showed that the titration method was equivalent to the sugar method, which is the reference one in EN 459-2. Interestingly, the comparison based on asphalt mixtures made with different aggregates showed that part of the hydrated lime was not fully recovered, because of the hydrated lime – aggregate reactions (Fig. 2). As a result, these reactions were more important for basalt aggregate (about 60% recovery), than moraine (about 80%) and limestone filler (about 90%).

2.3. Other methods

It is worth mentioning that Thermo-Gravimetric Analysis (TGA) could be also used in order to quantify $Ca(OH)_2$ in recovered filler.

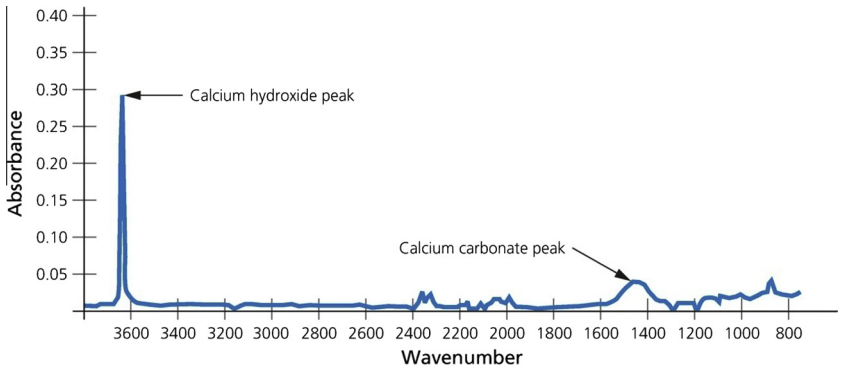

Fig. 1. FTIR spectrum of hydrated lime (absorbance in arbitrary unit and wavenumber in cm^{-1} – adapted from [14]).