



# Influence of low-temperature physical hardening on stiffness and tensile strength of asphalt concrete and stone mastic asphalt



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## HIGHLIGHTS

- Stiffness of asphalt mixes increased after isothermal storage at low temperature.
- No changes of tensile strength after isothermal storage were noted.
- Physical hardening of asphalt concrete and stone mastic asphalt was different.
- Two measures of physical hardening of asphalt mixes were proposed.

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## ABSTRACT

This paper presents laboratory testing of stiffness modulus and indirect tensile strength of three asphalt concrete (AC) and three stone mastic asphalt (SMA) mixes after isothermal storage at temperature of  $-20\text{ }^{\circ}\text{C}$ , at different time intervals up to 16 days. The tests under repeated dynamic loading showed physical hardening of all tested mixes which was manifested by an evident increase of their stiffness moduli after isothermal storage. Contrary to expectations, the tests on all mixes did not show any evident changes of the indirect tensile strengths which after isothermal storage remained almost constant, within normal scatter range. The differences in the physical hardening of asphalt concrete AC and stone mastic asphalt SMA were found. At the beginning of isothermal storage at  $-20\text{ }^{\circ}\text{C}$  the physical hardening was slower for the SMA. After 5–16 days of storage the SMA showed greater physical hardening than the asphalt concrete AC. Two measures which allowed the quantification of intensity of the physical hardening were introduced.

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## 1. Introduction

The physical hardening of asphalt binder is the term denoting the process of increase of stiffness of asphalt binder during extended storage at isothermal conditions. The process was first investigated in 1936 by Traxler and Schwyer [1] who studied different asphalt binders and observed substantial increase in their viscosities with time of storage at room temperature. They called this phenomenon “age hardening”. In 1937 Traxler and Coombs [2] explained the process of increase of viscosity of binders at isothermal conditions by the sol–gel transition. In 1950s Brown et al. [3] investigated several asphalt binders after isothermal storage and found reversible increase of their stiffness. They called the process “steric hardening” and explained it by the collapse of the molecular free volume at low temperature, isothermal sol–gel

transition and wax crystallization. The term “physical hardening” of asphalt binders was first used by Blokker and Van Hoorn [4] in 1959 who explained the process by wax crystallization and asphaltene aggregation. Struik [5] in 1970s tested the same process for several materials including polymers and some asphalt binders and called it the “physical aging” to distinguish it from the “chemical aging” which involves chemical reactions. In 1990 Pechenyi and Kuznetsov [6] explained the reversible hardening of asphalt binders during isothermal storage by the formation of partially ordered structures in the process of crystallization of asphalt material.

The physical hardening of asphalt binders at low temperatures, became the subject of intense research during last two decades, when it was noted that the process may have a significant impact on low-temperature behavior of asphalt pavements. It is known that physical hardening is related to the chemical composition and the source of asphalt binder base. Shrinkage and collapse of free molecular volume have been quoted as its prime cause [7,8].

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The molecular structuring and aggregation of asphaltenes and possibly crystallization of wax have been identified as additional factors responsible for the physical hardening [9–12].

The process of physical hardening of asphalt binders has been well documented by many researchers during the last two decades [7–10,13–15]. So far, much less attention has been paid to physical hardening of asphalt mixes. The question whether physical hardening occurs in asphalt pavement at cold temperatures and what is its importance in development of low-temperature cracking has been seriously disputed. Some researchers claim that the physical hardening in asphalt mixes can be cancelled by stress relaxation and does not affect low-temperature cracking of asphalt pavements [16,17]. Others [10–12,18–20] are of the opinion that the physical hardening is a very important factor and the asphalt mixes containing binders showing significant physical hardening at low temperatures are more susceptible to low-temperature cracking, what has been proven in field tests in Canada [11,20,21].

The dispute whether physical hardening occurs in asphalt mixes is caused by confounded results of laboratory tests, which were not always well designed to investigate this phenomenon. In most cases the thermal stress restrained specimen tests (TSRST) were used to test the physical hardening of asphalt mixes [14,22,23]. However, this method did not show a clear effect of physical hardening in asphalt mixes, which was the main basis to question the importance of physical hardening. A disadvantage of the TSRST test, when applied to the physical hardening, is that it does not measure directly the mix stiffness but rather the thermal stresses induced in a restrained asphalt specimen. The thermal stresses are in a very complex way related to several factors, such as elastic and viscous properties of the mix, its stress relaxation potential, thermal contraction properties of the mix, as well as specimen conditioning, rate of cooling and the thermal history. A better insight into physical hardening was obtained with a recently developed asphalt thermal cracking analyzer (ATCA) presented by Baglieri et al. [18], Bahia et al. [19] and Tabatabaee et al. [24]. The results from ATCA tests allowed the verification of the thermal stress model which included physical hardening [25].

There are very few published reports from testing of unrestrained asphalt concrete specimens, in which the stiffness modulus of asphalt mix was directly measured after isothermal storage at low temperatures [14,26]. In one of them [14], asphalt concrete specimens were tested in uniaxial tension–compression after storage at a low temperature and physical hardening was very evident. In another research [26], very small beams ( $6.25 \times 12.5 \times 102$  mm) cut from compacted asphalt concrete were tested in bending beam rheometer (BBR) according to AASHTO T313-06. In this case, the results were very much scattered, a part of the specimens showed physical hardening but a significant part did not.

Not only stiffness but also tensile strength is extremely relevant to the development of low temperature cracks in asphalt pavements. In the available literature on physical hardening there is no evidence that tensile strength of asphalt mixes was tested after extended storage at low temperatures. It was reported by El Hussein et al. [27], that micro-cracking developed in asphalt concrete samples during storage at low temperature, due to large differences between temperature contraction coefficients of asphalt binder and aggregate, which might lower tensile strength. Therefore, the tensile strength of asphalt mixes after prolonged storage at low temperatures is worth investigating.

## 2. Objectives

The main objective of this study was to determine effects of isothermal storage at low temperature on the stiffness and tensile strength of two types of asphalt mixes, namely asphalt concrete

AC and stone mastic asphalt SMA. Stiffness moduli and tensile strength of asphalt mixes were measured in indirect tensile test, after isothermal storage of specimens at  $-20$  °C, at different time intervals up to 16 days. The temperature of  $-20$  °C was selected for testing as it was a typical low temperature in Poland during severe winters. The second objective was to identify the influence of the mix type (asphalt concrete AC and stone mastic asphalt SMA), type of asphalt binder used, either neat and polymer modified, on the low-temperature physical hardening. The SMA is becoming the most popular wearing course on newly constructed main roads in Europe. The most research on physical hardening was carried out in the USA and Canada on asphalt concrete mixes and properties of SMA are less known. Therefore, part of this research was devoted to the low-temperature physical hardening of the SMA.

## 3. Material tested

In total, six asphalt mixes designed for wearing courses for medium and heavy traffic were tested for physical hardening. These included three asphalt concrete (AC) and three stone mastic asphalt (SMA) mixes. In order to obtain more realistic data the tested mixes were collected from asphalt plants and brought to the laboratory for testing. The mixes were produced by three different contractors and differed in mix composition, asphalt binders and aggregates used. The composition of tested asphalt mixes and types of aggregates used are presented in Table 1.

The mixes tested for physical hardening in this study represented all typical materials which have been used in Poland for wearing courses, including two types of mixes (AC and SMA), different asphalt binders (neat and polymer modified), two methods of polymer modifications (in refinery and in asphalt plant), different mineralogical types of aggregates, mix gradings and compositions. Testing of mixes sampled from asphalt plants had the advantage over laboratory produced mixes that real technological aging and normal scatter of mix composition were included. This gave the opportunity to assess the physical hardening in the realistic materials used for road construction.

Liquid adhesive agent (fatty amines) was added to all mixes to improve their water and frost resistance. In case of SMA the anti-dripping stabilizer in form of the cellulose fibers was also added.

Properties of asphalt binders are presented in Table 2. Three types of asphalt binders were used: neat, modified in refinery with styrene–butadiene–styrene (SBS) copolymer and modified with linear styrene–butadiene (SB) copolymer added in asphalt plant. All the binders were produced in the same refinery from the Ural crude oil. The binders contained low amount of paraffin. The harder grade 35/50 was produced with air blowing. The grade 50/70 was produced alternatively by compounding or by air blowing. The method of production of the base binder used for modification with the SBS polymer is unknown to the author. Asphalt binders were not tested in this study, but the data on their properties were provided by the refinery.

## 4. Testing of physical hardening of asphalt mixes

### 4.1. Preparation of specimens

Loose asphalt mixes collected from the asphalt plants were reheated, remixed and compacted in a gyratory compactor, to produce cylindrical specimens 100 mm in diameter and approximately 63 mm in height. It was expected that level of compaction and voids content in the compacted mix may affect physical hardening. Therefore, a great effort was undertaken to produce homogenous specimens. Greater number of specimens than required for testing of physical hardening was produced. Specimens whose voids differed more than 0.5% from the average value were rejected from further testing.

It was expected that besides the voids content also initial stiffness of asphalt mix might affect the process of physical hardening, in this way that stiffer specimens might harden during isothermal storage in different way than softer ones. To check homogeneity of mix stiffness all the specimens approved for testing of physical hardening were, before the proper test, examined in indirect tension modulus testing under repeated loading at  $+20$  °C, in the Nottingham Asphalt Tester (NAT), according to the European Standard PN-EN 12697-26 at 0.12 s time of loading pulse, frequency of 20 cycles per minute and vertical pressure of 220 kPa. Despite the

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