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Warm Mix Asphalt with Synthetic Zeolite: a Laboratory Study on Mixes Workability

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Abstract: In recent years, several warm mix asphalt technologies have been developed in order to enable significant energy savings and harmful emissions restraint by reducing mixing and compaction temperatures in hot mix asphalt production processes. This paper focuses on the evaluation of workability of WMA produced in laboratory by water-containing methodology with the addition of synthetic zeolite. Both volumetric and mechanical performance of warm asphalt mixtures compared with those of a reference hot mix were evaluated. For the Marshall specimens the variation of density with the compaction energy was also investigated. Finally, some considerations about the effect of the time of foaming (between mixing and compaction) on mixes workability are drawn.

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***Key words**: Asphalt workability; Compaction energy; Foaming process; Synthetic zeolite; Warm mix asphalt.*

Introduction

The objectives of preserving the environmental quality, reducing the use of natural resources and protecting human health, are fundamental in the aim of promoting the sustainable development.

Industrial activities produce the highest contribute to the greenhouse effects; for this reason the asphalt paving industry has continually strived to recycle more asphalt and to reduce emissions in order to become a more environment friendly industry [1-4]. The primary sources of emissions in an asphalt plant are the mixers, dryers, and hot bins, which emit particulate matter, such as dust, smoke, exhaust vapor, and other gaseous pollutants. Some other sources are the storage silos, truck loading operations, binder storage tanks, conveyers, stockpiles, etc. At the same time, the risk for the health of the workforce from asphalt fumes due to an excessive exposure during laying operations is high [5]. The bulk of CO_2, SO_2 and NO_x emissions from asphalt pavements happen during the initial construction, because of the high temperature required for mixing and paving [5]. Moreover, the whole process of asphalt mix production determines considerable energy consumption due to the high temperature reached in asphalt plants. That said, it is clear the need to develop innovative technology solutions in the asphalt industry that enable significant energy savings and a large emissions restraint by reducing mixing and compaction temperatures and maintaining, at the same time, high levels of performance without affecting the mechanical and functional properties of the mix [6-10]. In the last years, several new processes that reduce the usual temperature levels for Hot Mix Asphalt (HMA) have been developed [5, 11-12]. These technologies are referred to as Warm Mix Asphalt (WMA), an emerging class of asphalt mixture that reduces heating requirements during production and compaction operations. Several WMA technologies, based on different production processes, are been widespread worldwide. A brief description of them is reported in the subsequent section.

Warm Mix Asphalt Technologies.

WMA requires lower production and compaction temperatures if compared to HMA while aiming to maintain the desired performance. This spread is estimated between about 20 and 50℃, depending on the adopted technology [5-13]. WMA technologies are frequently classified by the methodology used to improve asphalt concrete workability [5-13]. In particular, two main categories can be identified as follows (see Fig. 1): (i) *foaming processes*; (ii) *addition of dopes*.

The foaming process is characterized by the injection of small amounts of water directly into the mix chamber or into the hot binder; in fact, when the water is dispersed in hot asphalt, it vaporizes (from contact with the hot asphalt) and it results in a binder expansion with a consequent reduction of the mix viscosity [14]. This process can be reached by means of the following sub-methodologies:

- Water-containing: this method uses synthetic zeolite to produce the foaming process. This type of additive is composed of aluminosilicates of alkali metals, and has been hydro-thermally crystallized. Zeolite's structure has large and interconnected spaces, which can accommodate a wide variety of cations (Na^+, K^+, Ca^{2+}, Mg^{2+}) and even molecules such as water. The spaces are interconnected and form long wide channels of varying sizes depending on the mineral. These channels allow the easy movement of the resident ions and molecules into and out of the structure. In particular, this absorption and desorption process through the zeolite interconnected cavities does not damage the crystal structure. When the temperature goes up over 80-90°C, zeolite structure releases the water crystallized, making the foaming effect to be reached [11, 13, 15].
- Water-based: this technology uses special nozzles to produce the foaming effect, injecting directly water under pressure into the hot binder flow. The water evaporation produces a large

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volume of foam that slowly collapses. Two classes of methodologies belong to this sub-category [13]: Foaming Binder and Two-Component Binder System. This last one uses a soft binder and a hard foamed binder at different times during the asphalt concrete production.

- Low Energy Asphalt: this method uses wet fine aggregate. Coarse aggregate is heated and mixed to the binder at the usual temperature and then it is mixed with the cold, wet fine aggregate. The moisture produced triggers the asphalt foaming process.
- Low Emission Asphalt: it is a combination of chemical and foaming technology. The binder containing a chemical additive is added to the hot coarse aggregates, and in the second phase, wet sand is added, which creates a foaming action [13].
- The methodologies based on the addition of dopes can be classified into two sub-categories depending on the dope's nature (organic or chemical). Organic dopes can be natural or synthetic wax. These additives modify the temperature viscosity curve of the binder, specifically in a temperature range above 100℃, where mixing and placement normally occur. The wax must have a melting point higher than the expected in-service temperature in order to avoid permanent deformations of the laid mix and to minimize asphalt distress at low temperatures [5-13]. Three main processes pertain to this technology, with respect to the type of the organic wax used:
- Fischer-Tropsch wax: this is a method for the synthesis of hydrocarbons and other aliphatic compounds from synthesis gas (CO/H_2). The difference between naturally occurring bituminous waxes and F-T waxes resides in their structure and physical properties. In particular, these waxes are long-chain aliphatic hydrocarbon waxes characterized by good oxidation and ageing stability, and can be stored indefinitely [5-13].
- Fatty acid amide: Amide waxes are synthetic fatty acid amides. They are manufactured synthetically by causing amines to react with fatty acids. The form crystallites in the bitumen, thus increasing asphalt stability and deformation resistance [5-13].
- Fossil fatty acid esters, also known as Montan wax: it is extracted from special waxy lignite. Since the melting point of this wax in its pure state is approximately 75℃, it is often blended with materials with a higher melting temperature such as amide waxes [5-13].

Finally, the methodology based on the addition of chemical dopes uses a combination of emulsification agents, surfactants, polymers, and additives to improve coating, mixture workability, and compaction, as well as adhesion promoters (anti-stripping agents). Additives are mixed with bitumen before adding it in the asphalt mixer.

Additivation with Synthetic Zeolite: a Literature Review

One of the most common WMA techniques (belonging to the group of the foaming process methodologies) is the addition of synthetic zeolite to the asphalt mix; it is widely used thanks to the easiness of the additive storing and handling operation. Furthermore, conventional mix plants do not endure particular changing because the synthetic zeolite, available in granular form, can be easily added

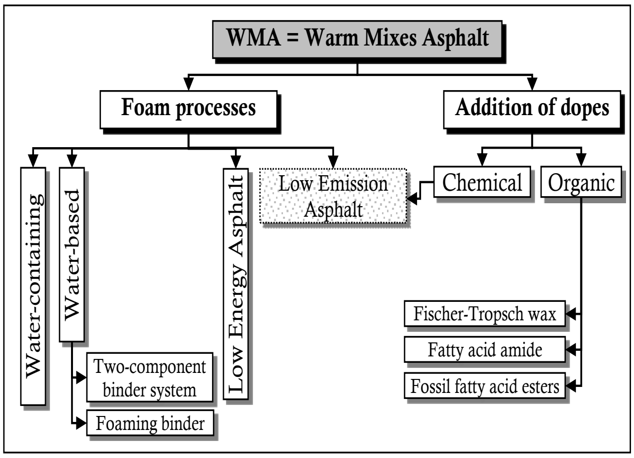

Fig. 1. Scheme of Warm Mixes Methodologies.

to the mix by means of an external dosage device. Very low quantities of material (about 0.3% on the total aggregates weight) are used in the mixing process.

The first major laboratory study on zeolites was performed by Hurley and Prowell [16]. They found that the addition of Aspha-min® zeolite reduces air voids by 0.65%, compared to the relative control mix; it does not affect the resilient modulus of the mix; it does not increase the rutting potential of mixes, though the rutting potential increase as mixing and compaction temperature decrease (which is the point of warm asphalt); it may increase the potential for moisture susceptibility [1, 16].

Other studies [17-18] evaluated the effect of zeolite on the performance of the asphalt binder (virgin/rubberized, aged or not). Gandhi et Amirkhanian [18] investigated on specific warm asphalt binder properties such as viscosity and rutting resistance ($G^*/\sin\delta$), measured by means of the rotational viscometer and the dynamic shear rheometer, (DSR) respectively. The results of this study revealed that zeolite did not significantly affect binder viscosities at 135℃ and 120℃: a very slight initial decrease was observed during the first 60 minutes after mixing due to the foaming of the asphalt. This effect vanished after about 60 to 90 minutes, when zeolite-modified binders exhibited viscosities significantly higher than the base binders.

Other researches confirmed these results [19-20] also for rubberized binders, attributing the increasing in the viscosity to the fact that the zeolite (added in fine powder form) acts as a mineral filler in the mix and after initial foaming it remains un-dissolved in the mix. In reality, the zeolite creates a very fine water spray when the entrapped water is released causing a volume expansion which lead to a better mix workability and compactability.

Akisetty et al. [19] investigated also on the effect of ageing on warm rubberized binders showing that the addition of the inorganic additive Aspha-min® determined a better resistance to permanent deformation at higher temperature than the control rubberized binders.

A detailed study on WMA water sensitivity was conducted by De Visscher et al. [21] by measuring the indirect tensile strength (ITS) before and after specimens conditioning in water, according to EN 12697-12 [22]. Results show that both ITS and ITS-ratio of warm mix asphalt with 0.3% of zeolite are similar to the values obtained for the reference hot mix, up to temperature reduction of 30°C. Higher temperature differences have negative effects on the water sensitivity. The results obtained for the studied mixes confirm that a reduction of temperature by 30°C is possible when using zeolites without affecting asphalt mix performance. No significant differences between warm and hot mixes were also observed regarding the behavior at low temperature (Thermal Stress Restrained Specimen Test, TSRST). D'Angelo et al. [5] confirmed these findings as well, demonstrating that all types of WMA are either equal to or better than the control HMA pavements in terms of resistance to thermal cracking, based on short-term field thermal cracking performance [14]. They also found that the addition of Aspha-min ® do not change the equivalent fatigue cracking if compared to traditional HMA pavements. These results are based on field short-term pavement performance evaluations in France, Germany and Norway [14].

In the study pursued by Aschenbrener et al. [23] the surface performance of WMA produced with Advera® zeolite were evaluated in terms of sand patch height (HS), according to ASTM E965 [24]. The results were very consistent between the HMA control and its associated WMA test section. The WMA test sections performed equally as well as the HMA control sections. Based on visual observation, all the HMA control and WMA test sections performed very well with respect to raveling and weathering [23].

The effects of the zeolite addition on mix macrotexture are strictly related to the warm mix higher compactability and to the lower residual air voids content of the laid pavement [25].

Several previous studies show the positive effect of zeolites on mix workability and compactability, regardless the compaction technique used (impact, gyratory and vibratory compaction) [26]. The controlled foaming effect of the addition of zeolite leads to a slight increase in binder volume, allowing asphalt binder to adequately coat the aggregate during mixing at lower temperatures. A better mix workability corresponds to a reduction in air void content, with a consequent influence on the surface texture performance.

The workability of warm mixes produced with zeolites depends also on the time elapsed between mixing and compaction operations [11-21]. In particular, the effect of foaming due to the addition of the additive in the mix is characterized by an optimum reaction time [19] during which the volume expansion reaches its maximum value. De Visscher et al. [21] observed an increase in compactability of laboratory samples, up to one hour of foaming time. The effect of zeolite, in fact, gradually vanishes when the mix is stored before compaction and the binder viscosity also increases. For this reason, it is steel necessary to better understand the relationship between the ease of compaction of warm mix asphalt and the reaction time of the additive used, depending on the compaction temperature.

Objectives and Scope

The paper focuses on the evaluation of workability as a consequence of compactability of WMA produced in laboratory by water-containing methodology, with the addition of synthetic zeolite. The main aim of the study is the identification of a maximum degree of mixes compactability as a function of a "Foaming Time" (FT), in which the additive can completely release the water contained, maximizing its foaming effect.

The consequence of the foaming effect is evaluated in terms of air voids content; the variation of this parameter in relation to the foaming time was also investigated, in order to identify the FT value that minimizes the percentage of voids in the warm mix.

The influence of this period of time, elapsed between mixing and compaction operations, on mixes workability was analyzed by means of a systematic comparison between traditional Hot Mix Asphalt (HMA) and Warm Mix Asphalt (WMA) produced at lower mixing and compaction temperatures.

Moreover, if the zeolite contained in the mix does not completely evaporate during mixing, because of the low mix temperatures, water may be left in close contact with the aggregate surface causing the cohesive failure of the binder. Therefore, additional test samples were produced and tested in accordance with the EN 12697-23 [27], Indirect Tensile Strength Test, ITS.

All experimental tests were performed in the Road Material Laboratory of the Department of Civil Engineering (formerly Territorial Planning), University of Calabria, Italy.

Materials and Methods

Limestone aggregate with a nominal maximum aggregate size of 15 mm was used for both WMA and HMA mixes. The main aggregate properties are listed in Table 1.

The aggregate gradation is a commonly used granulometric distribution for top layers according to Italian Standard specifications [31] (see Fig. 2). It should be noted that the design aggregate gradation is an average distribution between the upper and lower specification limits for surface courses [31]. The same

Table 1. Main Coarse Aggregate Properties.

Test/ Index	Value	Standard/ References
Aggregate nature	Limestone	-
Los Angeles Abrasion Test [%]	22.30	EN 1097-2 / [28]
Shape Index [%]	28.30	EN 933-4 / [29]
Aggregate specific gravity [g/cm^3]	2.788	EN 15326 / [30]

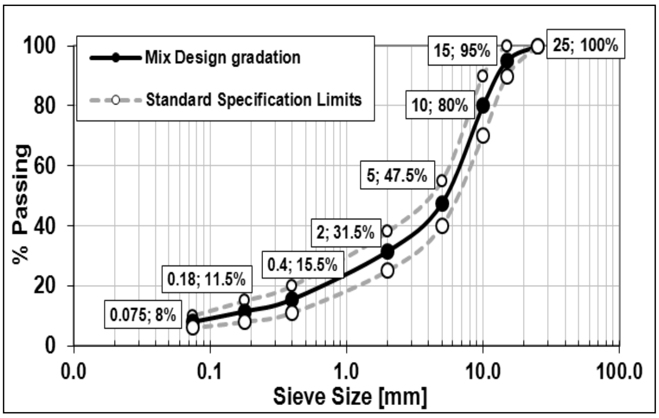

Fig. 2. Aggregate Gradation for both HMA and WMA Mixes.

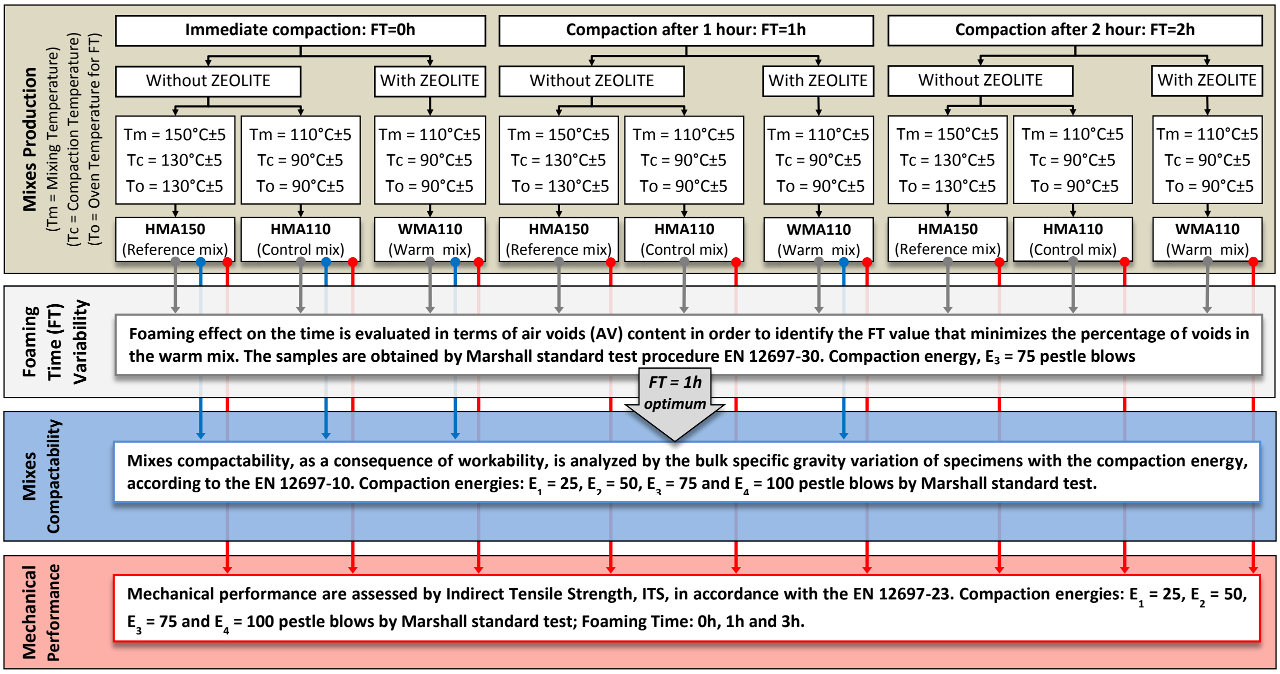

Fig. 3. Experimental Plan.

aggregate gradation was selected for both HMA and WMA mixes.

All samples, produced by Marshall standard test procedure (EN 12697-30 [32]), were obtained with a 50/70 penetration grade bitumen in a percentage of 5% by mass on the aggregate weight. A quantitative of Aspha-Min® zeolite of 0.3% on the aggregate mass was added to the mixtures, according to the manufacturers' recommendations [33]. The laboratory study carried out involved several parameters to be investigated; a detailed experimental plan was designed and carried out, see Fig. 3.

The first step of this research was the design and production of three types of mixes, two HMA and one WMA at two different mixing temperatures: 150°C and 110°C.

In particular, HMA mixed at 150°C (HMA150) is adopted as reference mix.

Two different values of foaming time (1 and 2 hours) were set: the mixes were stored in a thermostatically controlled oven at a temperature of 20 degrees lower than the mixing temperature. A condition of immediate compaction after mixing operations was also analyzed (Fig. 3).

The HMA 110 control mix, prepared at the same reduced temperature as the warm mix, was produced for a systematic comparison between WMA and HMA volumetric and mechanical performance; the reference hot mix (HMA 150) was also designed in order to investigate on the potentialities warm mix compared to a traditional asphalt concrete compacted at conventional higher temperatures.

Experiments and Results

Volumetric Properties of Mixes

All samples were produced following the Marshall standard test procedure (EN 12697-30 [32]). For the study of foaming time variability, the compaction energy was set on 75 pestle blows. Six Marshall samples were produced for each mix (both HMA and WMA with or without zeolite) at each foaming time, for a total of 54 specimens. As it is possible to see in Fig. 4 the effect of the additivation with zeolite on mix workability is higher after a storage time of 1 hour, when the WMA110 air voids content reaches a value lower than the one estimated for the HMA150 mix. In particular, for a FT value of 1 hour, the rate of decrease in the voids of the WMA110 (AV≈4.0%) is about 25% in relation to the reference mix (HMA150, AV≈5.4%) and it reaches a value of about 60% if compared to the hot mix compacted at the same temperature (HMA110, AV≈9.75%). In absolute, the WMA110 mix compacted after 1 hour has a value of air voids (AV≈4.0%) comparable with the HMA 150 mixture (AV≈ 3.30%) in a condition of immediate compaction (FT=0h).

The effect of foaming seems to be less visible after two hours of storage in the oven when the difference in air voids content of WMA110 (AV≈8.65%) and HMA110 (AV≈8.70%), mixes compacted at 90°C, is quite negligible (Fig. 4).

Only for mixes HMA150, HMA110, WMA110 (immediate compaction, FT=0h) and WMA110_1h (storage time of 1 hour, FT=1h), mix compactability was analyzed by calculating the variation of specimens density (by the parafilm method, ASTM D1188-07 [34]) with the compaction energy, according to the EN 12697-10 [35]. Four different compaction energies were set: 25, 50, 75 and 100 pestle blows; six Marshall samples were produced for each mix at each number of pestle blows, for a total of 96 specimens.

The compaction energy is expressed in terms of number of pestle blows for each side of the specimen. The equation that governs the evolution of mixes densification has the following expression, Eq. (1) [34]:

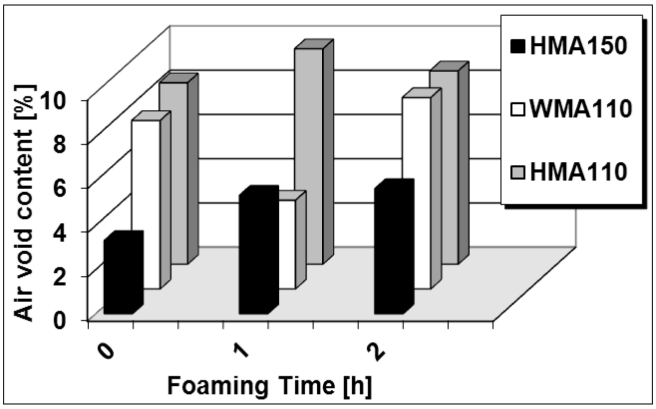

Fig. 4. Air Voids Content Variation with Foaming Time.

Table 2. Densification Parameters According to EN 12697-10 [35].

	ρ_∞ [g/cm^3]	ρ_0 [g/cm^3]	C	R
HMA150	2.61	2.24	51.4	0.14
HMA110	2.61	2.21	68.8	0.15
WMA110	2.61	2.21	73.6	0.15
WMA110_1h	2.61	2.14	53.4	0.18
ΔC%	HMA150h	HMA110	WMA110	WMA110_1h
HMA150	0%	-34%	-43%	-4%
HMA110	\	0%	-7%	22%
WMA110	\	\	0%	27%
WMA110_1h	\	\	\	0%

$$\rho(E_1) = \rho_\infty - (\rho_\infty - \rho_0)\exp\left(\frac{E_1}{C}\right) \quad (1)$$

where: $\rho(E_1)$ is the density of the specimen at the compaction energy E_1 (Mg/m^3); ρ_∞ is the maximum density of the specimen in an ideal condition of no residual air voids (Mg/m^3); ρ_0 is the estimated initial density of the specimen (Mg/m^3); C is the resistance to compaction (it express the mix workability); E_1 is the energy applied on the specimen (for impact compaction it represents the number of pestle blows). The values of ρ_∞, ρ_0 and C can be calculated by using the least square method, minimizing the squared difference between theoretical and experimental data. The lower C is, the higher mix workability will be. Furthermore, in Table 2, ΔC% represents the percentage difference between the values of compaction resistance (C) among all mixes.

Starting from the values of ρ_∞ and ρ_0 it is possible to evaluate another parameter related to mix to workability, named *relative compaction potential*, by means of the Eq. (2):

$$R = \frac{(\rho_\infty - \rho_0)}{\rho_\infty} \quad (2)$$

More detailed information about the densification parameters estimated for all mixes are summarized in Table 2.

The variation of specimens density in relation to the compactive effort, expressed as the number of blows, is shown in Fig. 5. These results are the average values of six measurements for all mixes.

The analysis of the densification parameters shows that the mix produced with the addition of zeolite at 110°C with a foaming time of 1 hour has a value of resistance to compaction similar to that

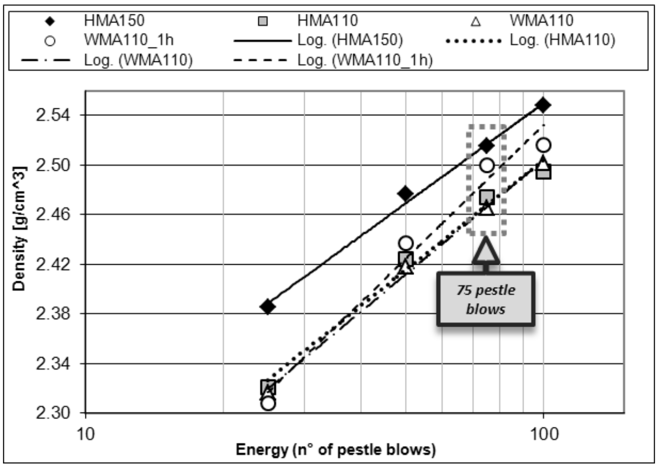

Fig. 5. Density Variability with Compactive Effort.

estimated for the reference mix HMA150, confirming that the zeolite needs for a period of time to maximize its foaming effect in the mix (ΔC% ≈ -4%).

The rate of increase of the warm mix density against the increase in compactive effort is more visible for a compaction energy of 75 pestle blows (Standards common value for mixes compaction) when the behavior of the WMA110_1h mix is similar to that of the reference HMA150 mix. The densification process at low energy values (25 pestle blows) is similar for all mixes compacted at a temperature of 90°C, with and without the additive (Fig. 5).

A slight difference (ΔC% ≈ -7%) between the C values registered for the WMA110 and the HMA110 was observed (see Table 2), confirming that the additive improves mix workability also in a condition of immediate compaction if compared with a hot mix produced at lower temperatures than the conventional asphalt-mixing process. Comparing the compactability values of both WMA110 and HMA110 mixes to the ones estimated for the HMA150 mix it is possible to highlight an increase in mix resistance to compaction (ΔC%) of about 43% and 34%, respectively (see Table 2).

As regards the relative compaction potential, R (see Table 2), the lowest value was registered for the HMA150 mix: the higher mixing and compaction temperature allow the mold to reach a higher initial density because of the lower binder viscosity. Moreover, in a condition of immediate compaction, also the HMA110 and the WMA110 mixes have quite similar values of R if compared to the reference hot mix HMA150; the difference of about 40°C in mixing and compaction temperature leads to an increase in R from 0.14 to 0.15, probably because the samples compacted immediately after mixing are characterized by a thermal gradient with a no-uniform temperature distribution. The effective temperature of the specimens may be higher than the fixed compaction temperature of 90°C. On the contrary, when the WMA110_1h mix is stored in the oven for 1 hour, a temperature stabilization process occurs: the whole mold is characterized by an effective temperature of 90°C (uniform distribution) that determines higher values of the compaction potential (R = 0.18, see Table 2).

Mechanical Performance of Mixes

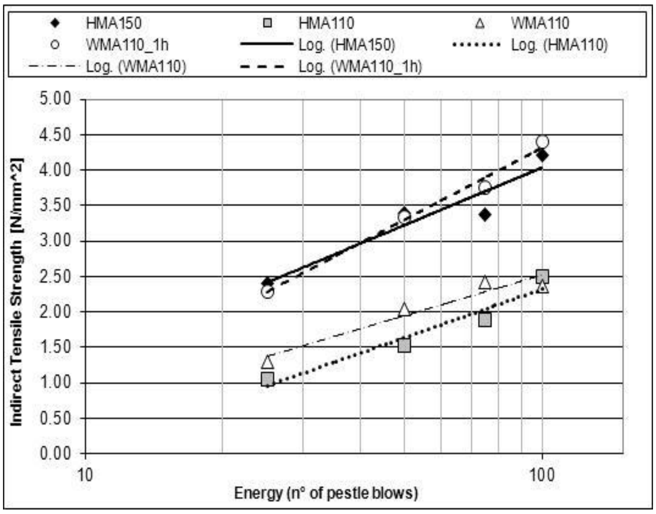

Fig. 6. Indirect Tensile Strength Variability with Compactive Effort.

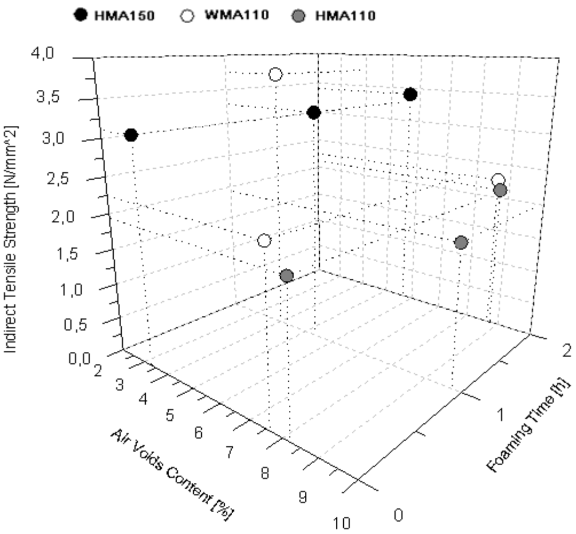

Fig. 7. Indirect Tensile Strength Variability with Air voids Content and Mix Type.

Mechanical performance were assessed in terms of Indirect Tensile Strength, ITS, in accordance with the EN 12697-23 [27], as it is shown in Fig. 6. Mechanical results highlight that the ITS values of the warm mix compacted after 1 hour of foaming time are very close to those registered for the HMA150 reference mix. In accordance with the volumetric results the foaming effect of the additive leads to a better mix workability that contributes to a reduction of air voids content.

It seems to be evident that the addition of the synthetic zeolite does not influence the test results. The small amounts of water released in the foaming process have no negative consequences on the binder-aggregate adhesion, because the main phenomenon that contributes to an increase in tensile strength is the better mix compactability.

It is also observed that ITS decreases with decreasing the compaction temperature from 130°C to 90°C in a condition of immediate compaction, regardless the compactive effort, which may be due to the increasing in air voids content. Fig. 7 is a synthesis of both mechanical and volumetric results: mixes with higher compactability (lower C values, see Table 2) and, consequently, lower air voids contents are characterized by higher values of ITS.

Conclusions and Recommendations

This paper presents the results of a laboratory study aimed to investigate on both volumetric and mechanical performance of warm asphalt mixes produced with the addition of the Aspha-min synthetic zeolite in a quantitative of 0.3% on the total aggregate weight. WMA performance were systematically compared to those of a reference hot mix asphalt (mixing temperature of 150°C and compaction temperature of 130°C) and of a control hot mix compacted at the same lower temperature selected for the warm mix (90°C).

A deep investigation about the effect of the foaming process on mix workability was also carried out in order to evaluate if the additive needs for a reaction time to completely release the entrapped water maximizing the foaming effect. Starting from the idea that the additive can improve its performance into the mix during a period of time elapsed between mixing and compaction operations, three different sets of mixes were produced:

- immediate compaction after mixing: two HMA compacted at 130° (reference mix) and at 90°C (control mix) and a WMA compacted at 90°C;
- 1 or 2 hours of foaming time (FT): the same mixes were compacted after 1 and 2 hours of storage in a controlled oven, respectively.

Mixes compactability was firstly evaluated in terms of air voids content variation in relation to the foaming time. Results showed that the foaming effect of the zeolite was higher after a storage time of 1 hour, when the air voids content measured for the warm mix reached a value lower than the one estimated for the reference HMA mix produced at 150°C. The effect seems to vanish after two hours of mixes storage in the oven.

The analysis of the densification parameters calculated according to the EN 12697-10 confirmed the first volumetric results, showing that that the mix produced with the addition of zeolite at 110°C with a foaming time of 1 hour had a value of resistance to compaction similar to that estimated for the reference mix HMA150. The variability of specimens density in relation to the compactive effort, expressed as the number of pestle blows, was evaluated by means of the densification curves analysis (EN 12697-10). The highest values of density for the warm mix compacted after 1 hour were reached for a compactive effort of 75 pestle blows.

Mechanical performance were assessed in terms of Indirect Tensile Strength, ITS, in accordance with the EN 12697-23. Results showed that the ITS values of the warm mix compacted after 1 hour of foaming time are very close to those registered for the HMA150 reference mix. It is possible to conclude that the small amounts of water released in the foaming process have no negative consequences on the binder-aggregate adhesion because the increase registered in tensile strength is primarily due to a better mix compactability.

It is important to observe that these results are related to a specific type of compaction (Marshall impact compaction).

For this reason, further investigations are needed to generalize this findings, regardless the laboratory compaction technique used.

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