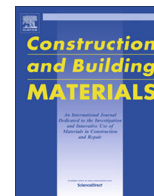




Contents lists available at ScienceDirect

Construction and Building Materials

journal homepage: www.elsevier.com/locate/conbuildmat

Properties of the Warm Mix Asphalt involving clinoptilolite and Na-P1 zeolite additives

Agnieszka Woszuk^{a,*}, Wojciech Franus^b^aLublin University of Technology, Faculty of Civil Engineering and Architecture, Department of Roads and Bridges, Nadbystrzycka 40, 20-618 Lublin, Poland^bLublin University of Technology, Faculty of Civil Engineering and Architecture, Department of Geotechnical Engineering, Nadbystrzycka 40, 20-618 Lublin, Poland

HIGHLIGHTS

- New direction of using the Na-P1-type synthetic zeolite produced from fly ash.
- NaP1-type synthetic zeolite and natural zeolite addition significantly decreases the mix asphalt compaction temperature.
- Soaking of the zeolite minerals with water allows to decrease the amount of zeolite d to mix asphalt.

ARTICLE INFO

Article history:

Received 9 December 2015

Received in revised form 25 March 2016

Accepted 29 March 2016

Keywords:

Warm Mix Asphalt

Natural zeolite

Synthetic zeolite

Gyratory compactor

Stiffness modulus

Wheel-tracking test

Water sensitivity

ABSTRACT

The use of Warm Mix Asphalt (WMA) resulted in road construction being more environmentally friendly and more economical. One of the WMA production methods is asphalt foaming through addition of different modifiers, including zeolites. In this study, a possibility of decreasing the compaction temperature of AC 16 W 35/50 WMA was analyzed. The applied additives included: NaP1-type synthetic zeolite obtained from fly-ashes and natural zeolite – clinoptilolite. Both zeolite materials prior to the process of asphalt foaming were soaked with water. In the conducted compactibility tests in a gyratory compactor, the optimal of zeolite additive was established, which included 1% clinoptilolite, 0.5% NaP1 and 0.4% of both zeolites soaked with water w/m. For mix asphalt with optimal zeolite amount, tests of air void content, stiffness modulus, water sensitivity and resistance to rutting were performed in different compaction temperatures.

© 2016 Published by Elsevier Ltd.

1. Introduction

Modern road surface construction technologies are becoming a part of the world-wide pro-ecological trend. They provide durability of the surface, comfort and safety of the users, and at the same time, they are environmentally friendly and economical. These technologies include mix asphalt with lowered production and compaction temperature. While the production temperature of traditional Hot Mix Asphalt (HMA) mixes is 140–200 °C depending on the asphalt type [1], the Warm Mix Asphalts (WMAs) allow to reduce the technological temperatures by 20–40 °C [2–6]. Currently, there are over 20 known technologies which allow to decrease the mix asphalt production and compaction temperature [2,7]. The beginnings of WMA use included application of foamed

asphalt [8]. Asphalt foaming technologies can be divided into two groups [2,7,9]:

- water-based
- water-containing.

Foaming is based on the addition of water with a temperature of 20 °C into the hot asphalt (temp. 170–180 °C) or into the mix chamber. Then, the created water vapor foams the asphalt over the course of a few dozen seconds. A new asphalt foam, with low viscosity and large specific surface area which provides appropriate aggregate coating at lower temperature [10]. After a few minutes, the water vapor disappears and asphalt recovers its initial properties. The optimal amount of added water ranges between 2 and 4% with regard to the asphalt mass [11–13]. Too high amount of water may result in washing the aggregate out of the binder [14]. Despite the real possibility of decrease technological temperatures, high cost of adapting asphalt plants for asphalt foaming is a serious barrier preventing the spread of this technology.

* Corresponding author.

E-mail addresses: a.woszuk@pollub.pl (A. Woszuk), w.franus@pollub.pl (W. Franus).

Another asphalt foaming method is application of zeolites. Zeolites are aluminosilicates with a structure which includes empty spaces called pores and channels. A characteristic feature of zeolites is the ability to accumulate the so-called zeolite water in the channels of their crystal structure, which can be removed through heating in the temperature range of 100–400 °C and then absorbed again when the humidity rises or it can be substituted by other substances. Such a specific internal structure provides zeolites with many physical and chemical features which are particularly advantageous for different kinds of industrial applications [15–17]. To the group of natural zeolites belong about 100 minerals. The most important zeolites present in natural deposits include: clinoptilolite, chabazite, mordenite ([18]). Synthetic zeolites can be obtained from chemical reagents in chemical reactions of sodium silicate and sodium aluminate, mineral materials (clay minerals, silica-group minerals) and some waste byproducts of carbon burning (such as fly ash) [19,20]. Both synthetic zeolites, as well as natural ones, are diversified in terms of internal structure and physicochemical and surface properties.

Asphalt foaming using zeolites is possible due to gradual release of water contained in their mineral structure. The release of zeolite water does not occur in a sudden manner, but is a long-term process, thus the viscosity decrease and workability improvement are possible during the mix asphalt production, placement and compaction [4]. Performed Tests of mix asphalt with zeolite addition indicated that the best compactability was achieved after 1-h of conditioning and Foaming effect disappeared after 2 h [21]. The majority of WMA technology studies with synthetic zeolites so far comprises the 0.3% w/m effect of Aspha Min zeolite addition (Eurovia GmbH, Germany) [21–24] and 0.25% w/m of Advera (PQ Corporation, USA) [25,26]. These zeolites contained about 21 percent of water (by mass) which was released in the temperature range of 85–180 °C [27,28]. Studies of asphalt and mix asphalt with a different quantity of synthetic zeolite additive were also conducted [29–32], as well as those using natural clinoptilolite zeolite [31–33]. Because the mix asphalt production technology with zeolite addition does not differ from technologies used traditionally in mix asphalt production [34,35], it is possible to introduce it without incurring significant financial outlays.

2. Objectives and scope

The main aim of the study is to assess the possibilities of applying natural zeolite (clinoptilolite) and synthetic one (NaP1) and their variations modified through soaking with water for production of mix asphalt with decreased compaction temperature. Optimal estimation of the zeolite amount and appropriate compaction temperature was defined using compactability tests in gyratory compactor. In the next stage, mechanical properties of reference mix asphalt and mix asphalt with zeolite addition were evaluated in different compaction temperatures. The flow chart of performed tests is presented in Fig. 1.

3. Materials used and mix design

The base bitumen with a 35/50 penetration grade was obtained from LOTOS Asphalt Sp. z o.o. The following tests were performed in order to characterize the base bitumen properties: penetration (EN 1426:2009), breaking point (EN 12593:2009), ring and ball softening point (EN 1427:2009) and viscosity (ASTM D 4402). Based on the results of penetration and softening point tests, penetration index (EN 12591:2010) defining the thermal vulnerability of asphalt was calculated [36–40]. The test results are presented in Table 1.

The aggregates used in laboratory tests were provided by Przedsiębiorstwo Robót Drogowych Sp. z o.o. Zamość. Particle size distribution

was assessed using dry sieving method (EN 933-1:2012) [41], mineral filler particle size distribution was assessed using air jet sieving method (EN 933-10:2009) [42].

The gradation of the aggregates is given in Table 2. The mineral mix composition was designed using grading envelope method.

Subthreshold values apply to Technical Requirements effective in Poland [43].

In the mineral composition of granodiorite the main components were quartz, soda-lime feldspars (plagioclases) and calcium feldspars (orthoclase) which are accompanied by minerals from a group of micas (biotite, muscovite) and pyroxenes. Granodiorite was acquired from Vyryvskij Karjer deposit (Ukraine).

Dolomite rock consisted mainly of a mineral dolomite $\text{CaMg}(\text{CO}_3)_2$ and calcite CaCO_3 . The rock used for the mix production was acquired from the Dolomite Mine Piskrzyń (Poland).

The limestone was composed of calcite, accompanied by trace amounts of dolomites, clay minerals and quartz. The limestone was acquired from Mine Siatkówka Nowiny (Poland).

Zeolites used in this study represent various structure topologies. Na-P1 has a gismondine-like structure (GIS) where two 4-membered rings form an 8-membered intersecting channel of $3.1 \times 4.5 \text{ \AA}$ and 2.8 and 4.8 \AA along [100] and [010] directions, respectively. Natural clinoptilolite has a heulandite topology (HEU) with two-dimensional channel system consisting of 10-membered (tetrahedron) rings sized $5.5 \times 3.1 \text{ \AA}$ and 8-membered rings sized 4.1×4.1 along [001] and $3.4 \times 2.8 \text{ \AA}$ along [100] [44].

The synthetic zeolite was synthesized in the hydrothermal reaction of fly ash from hard coal combustion with sodium hydroxide (NaOH). The fly ash for zeolite synthesis was supplied by the Kozienice Power Plant in Poland. The preparation of the zeolite Na-P1 was performed on the quarter technical scale using the patented installation for synthesis of zeolites [45,46]. The content of pure zeolite phase in the obtained product was about 70%.

Natural zeolite clinoptilolite (ZN-C) in a form of zeolitic tuff (composed of 75% of pure clinoptilolite phase, cristobalite, quartz, feldspar and clay minerals – montmorillonite and illite) was acquired from Sokyrnytsya deposit (Transcarpathian region, Ukraine).

Not only did the mineral structure differ zeolites used in the study but also the particle size distribution and the texture. Particle size distribution of the studied materials is presented in Fig. 2. Textural parameters of the zeolites used are presented in Table 3.

Na-P1 exhibited unimodal particle size distribution. In this material particles of the size around $25 \mu\text{m}$ constituted 55% of the volume. Particle size distribution of clinoptilolite was bimodal with peaks at 25 and $300 \mu\text{m}$, which constituted 19% and 23% of the volume, respectively.

The amount of water in dry zeolite Na-P1 was 28% and in clinoptilolite 17%. The tests were performed with zeolite addition in air-dry condition, as well as after incorporating of additional water to zeolite structure. The water saturation of zeolites in relation to dry mass was 75% and 25% w/m for Na-P1 and clinoptilolite, respectively. This amount of water was responsible for a loose form of the zeolite material which provided equal distribution of the additive in mix asphalt.

A Reference material included mix asphalt intended for binding layer of a road loaded with KR 3–4 category traffic (AC 16 W 35/50), designed in accordance with Polish technical standards [43]. The amount of dry asphalt was 4.6% in relation to the mix asphalt mass.

4. Test methods

4.1. Determination of compaction temperature and proper addition of zeolite

Determination of compaction temperature and the amount of dry zeolite was conducted in a compactability test in a gyratory

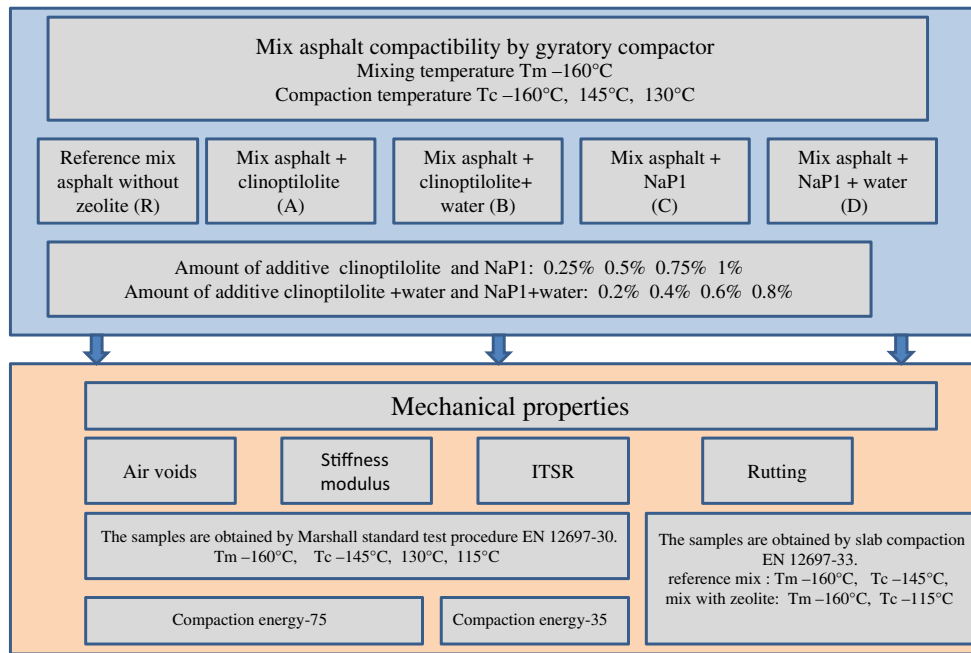


Fig. 1. Experimental Plan.

Table 1
Properties of the base bitumen.

Test	Specification	Result	Specification limits
Penetration (25 °C; 0.1 mm)	EN 1426:2009	36.5	35–50
Softening point (°C)	EN 1427:2009	55.80	50–58
Breaking point (°C)	EN 12593:2009	–14	<–5
Viscosity at (135 °C)- Pa s	ASTM D 4402	0.844	
Penetration index	EN 12591:2010	–0.56	–1.5 to +0.7

compactor (EN 12697-31:2007) [47]. Incline angle was assumed as 1.250, vertical load as 600 kPa, longitudinal axis rotation rate – 30 rotations/min, rotation number – 100. Hot mix prior to test was termostated in a form on a laboratory drier for 45–60 min. Compaction temperature of the reference mix was assumed based on the type of used asphalt – 160 °C [47]. Samples reference mix and samples with zeolite additions were compacted appropriately in temperature of 145 and 130 °C.

Conditions of sample preparation in Marshall hammer (height of 63.5 mm and diameter of 101.6 mm) were determined

according to the standard (EN 12697-30:2012) [48] and Polish technical requirements [48]. Before preparation of the first sample, the form was heated to compaction temperature. Compaction temperature of the reference mix asphalt was assumed based on the type of used asphalt – 145 °C [43]. Compaction temperature was decreased for subsequent samples by 15 °C to 115 °C. The samples for air voids test and stiffness modulus were performed using 75 hits per side, each. The samples for tests of Indirect Tensile Strength Ratio (ITSR) were made using 35 hits per side, each. Optimal zeolite addition to mix asphalt was assumed based on the results obtained from compactability tests in a gyratory compactor and included: 1% clinoptilolite, 0.5% for NaP1 zeolite and 0.4% w/w for water-soaked zeolites, respectively.

4.2. Air voids

Air voids content in compacted samples was calculated based on the EN 12697-8:2005 standard [49]. Polish requirements for the binding layer AC 16 W are within the limits of 4–7%.

Table 2
Gradation of the aggregates.

Test	Limestone 0/4	Granodiorite 4/8	Dolomite 8/11	Granodiorite 11/16	Mineral filler	Combined gradation (%)	Specification limits
Mixture ratio (%)	34	24	18	20	4		
Sieve size (mm)							
31.5							
22.4						100.0	100
16				100.0		97.6	90–100
11.2			100.0	88.0		80.0	70–90
8		100.0	87.0	12.0		60.4	55–85
5.6		91.0	3.0	1.0		47.5	
4	100.0	39.0	1.0	1.0		39.6	
2	99.6	7.0	1.0	1.0		30.6	25–50
1	79.3	1.0	1.0	1.0		20.0	
0.5	50.6	1.0	1.0	1.0		14.3	
0.25	35.2	1.0	1.0	1.0		10.7	
0.125	25.7	1.0	1.0	1.0	100.00	8.3	4–12
0.063	19.4	1.0	1.0	1.0	97.00	6.2	4–10
0.00	15.4	1.0	1.0	1.0	83.0	0.0	

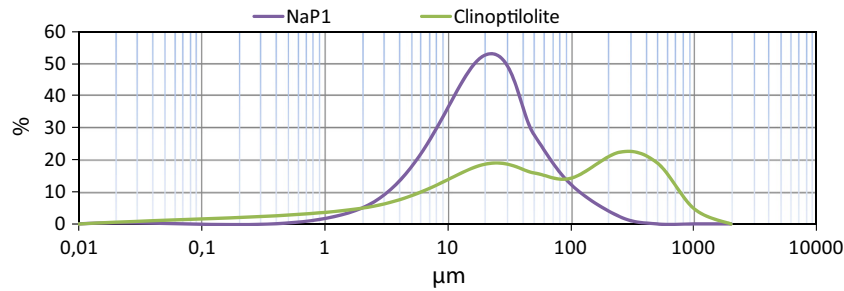


Fig. 2. Distribution of zeolite particle sizes.

Table 3

Textural parameters of clinoptilolite, zeolite NaP1 and mineral filler.

Materials	S_{BET} m ² /g	V_{mic} cm ³ /g	S_{mic} m ² /g	V_{mes} cm ³ /g	S_{mes} m ² /g	D_p nm
Clinoptilolite	18.3	0.0051	10.65	0.046	7.68	10.0
Na-P1	94.48	0.0048	10.62	0.233	85.86	8.9

S_{BET} – specific surface area, V_{mic}/V_{mes} – volume of micropores/volume of mesopores, S_{mic}/S_{mes} – surface of micropores/surface of mesopores, D_p – average pore radius.

4.3. Analysis of water sensitivity

The tests water and frost resistance of the concrete asphalts were performed based on the EN 12697-12:2008 standard [50] and Polish technical requirements [43]. This test evaluates the effect of one freezing cycle of saturated mix asphalt samples on indirect resistance to Marshall-type sample stretching. There were 8 samples prepared with every mix asphalt types and divided into two groups: 'dry set' and 'wet set' of similar heights and bulk densities. The control series samples were conditioned in a laboratory on flat surface in room temperature of 20 ± 5 °C. The procedure of conditioning wet samples with 1 cycle of freezing consisted of three stages:

Stage 1: The samples were exposed to vacuum water absorption for 30 min, in room temperature of 20 ± 5 °C, with absolute pressure of 6.7 kPa.

Step 2: The samples were exposed to prolonged effect of water in increased temperature by placing them in a water bath with the temperature of 40 °C for 68 h.

The third stage includes subjecting the samples to 1 cycle of freezing-defrosting.

The samples taken out of water bath were wrapped in "stretch"-type foil and placed in a plastic bag containing 10 ml of water. Tightly sealed bags with samples were placed in a cooling chamber with temperature of -18 °C for 16 h. The samples is placed in a bath with water temperature of 25 °C, where they are stored for 24 h. In this step, after defrosting, the samples are unwrapped. The samples from both sets were used to test the resistance to indirect tensile strength in Marshall's hammer, according to the EN 12697-23:2009 standard [51]. The temperature of sample testing from both sets was 25 °C. Based on the obtained results, an indicator of resistance to ITS_R was calculated:

$$ITS_R = \frac{ITS_w}{ITS_d}$$

where: ITS_R – Indirect Tensile Strength Ratio [%], ITS_w – the average strength for the wet samples [kPa], ITS_d – the average strength for the dry samples [kPa].

According to Polish technical regulation, minimum value of ITS_R indicator for the binding layer, KR 3–4, is 80%.

4.4. Study of stiffness modulus

The study of the stiffness modulus was performed according to the EN 12697-26:2012 standard [52]. The test was performed with

controlled tension. The samples of $\varnothing = 100$ mm and $h = 60$ – 65 mm were subject to five-time dynamic load applied to the sample vertically, along the diameter. Force increase time, measured from zero to a maximum value was 0.124 s. Maximum force generated a horizontal dislocation of sample equal to 5 μ m. Between the subsequent force impulses, there are 3-s long delays. The test result was calculated automatically by a control program, as an arithmetic mean from stiffness modulus for each of 5 force impulse measurements. The sample, after performing the test, was rotated by 90° around the horizontal axis and tested again. A reliable stiffness modulus for each sample was a mean average out of two measurements. The final result was the arithmetic mean out of 3 tested samples.

The tests were performed in three temperatures. 23 °C, 10 °C, -2 °C. These are the average temperatures for the Summer, Spring-Autumn and Winter periods in Poland. The following Poisson's ratios were applied for the temperatures, respectively: 23 °C – 0.4; 10 °C – 0.3; 2 °C – 0.25 [53].

4.5. Wheel-tracking test

The test of surface asphalt layer's resistance to rutting was performed in a small rutting tester according to A procedure, test in air (EN 12697-22:2008) [54]. The testing temperature was 60 °C, the number of cycles – 10 000. Testing samples of 300 mm \times 400 mm \times 60 mm were made using a laboratory slab roller compactor (EN 12697-33:2008) [55]. Prior to compaction process, the mix asphalt was conditioned in mixing temperature over 30–45 min. Reference mix compacting temperature was assumed based on the type of used asphalt – 145 °C. The compaction temperature of products with zeolite addition was 115 °C. The resistance to permanent deformation of the mix asphalt was defined by two parameters: wheel-tracking slope (WTS_{AIR}) and proportional rut depth (PRD_{AIR}).

5. Results and discussion

5.1. Determination of compaction temperature and optimum zeolite content

The results of compactability test in a gyratory compactor are presented in Figs. 3 and 4. The best compactability, expressed by the air voids, was noted for the addition of synthetic zeolite

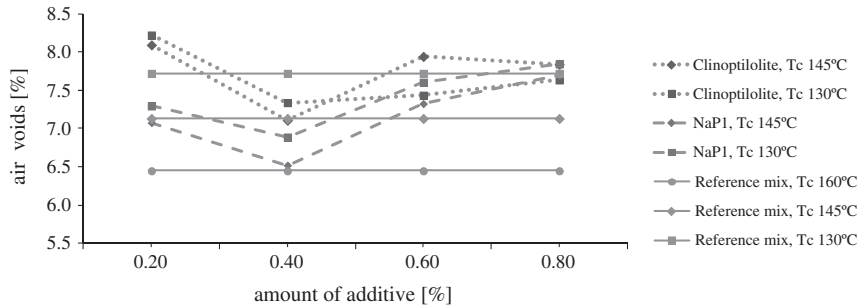


Fig. 3. Compactibility of samples with the addition of zeolites.

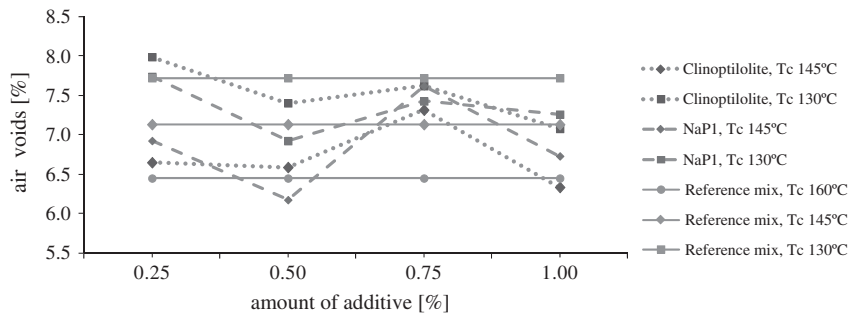


Fig. 4. Compactibility of samples with the addition of water-soaked zeolites.

NaP1. It was 0.4% for Na-P1 soaked in water w/m and 0.5% for NaP1 without soaking. The air voids decreased from 8.5 to 13% in relation to reference mix asphalt with the same compaction temperature.

In case of using 0.4% clinoptilolite soaked with water, a noticeable improvement in compactability could be noticed in compaction temperature of 130 °C. The addition of soaked zeolites exceeding 0.4% did not improve the mix asphalt compactability in both cases. The best improvement of the mix asphalt compactability with clinoptilolite addition was achieved with 1% addition.

Better compactability of mix asphalt samples with NaP1 synthetic zeolite addition than with clinoptilolite might result from textural properties of these minerals. Zeolite Na-P1 material had a 5-time higher specific surface area (94.48 m²/g) than clinoptilolite (18.3 m²/g) and was a more porous material (Table 3). Due to the higher textural parameters it was able to “absorb” more water and release it more at the same time, thus improving the asphalt’s foaming effect.

The obtained results indicated a possibility of decreasing compaction temperature by 15 °C. Compactability tests with synthetic zeolite addition of 0.3% w/m indicated the decrease in air void content in relation to control mix asphalt, with compaction temperature decreased to 40 °C [43]. The type of materials used for tests has an effect on the results’ divergence: both the zeolite type and the mix asphalt type.

According to performed compactability results, the most optimum zeolite content addition is: 1% for clinoptilolite, 0.5% for synthetic zeolite NaP1 and 0.4% of w/m water-soaked zeolites. Tests of mechanical properties were performed for mix asphalt with optimal zeolite amount.

5.2. Air voids

The results of air void content tests on Marshall samples indicated a minimal compactability improvement with regard to the reference mix asphalt after applying the zeolite addition. With the lowest compaction temperature (115 °C), the improvement in

compactability was the highest: air void decreased from 9% (NaP1 zeolite addition, NaP1 + water) up to 14% (clinoptilolite addition). Together with the decrease in compactability temperature, the air void content increased, regardless of the zeolite material type. Air void content in each mix asphalt was within the required limits [43] (Fig. 5).

On the basis of one way ANOVA analysis it can be concluded that the content of voids in the samples compacted at 115 °C is statistically significant regardless of the type of zeolite material. At this temperature the highest improvement in compaction has occurred. Obtained results indicate that compaction in Marshall compactor should not be the basis for the assessment of compactability of WMA with zeolite. The temperature of compaction had greater impact on void content than the type of added zeolite. Compaction tests should be performed in a gyratory compactor.

5.3. Analysis of water sensitivity

The results of indirect tensile strength test for each temperature and zeolite addition temperature combination are presented in Table 4, ITSR values (%) are presented in Fig. 5.

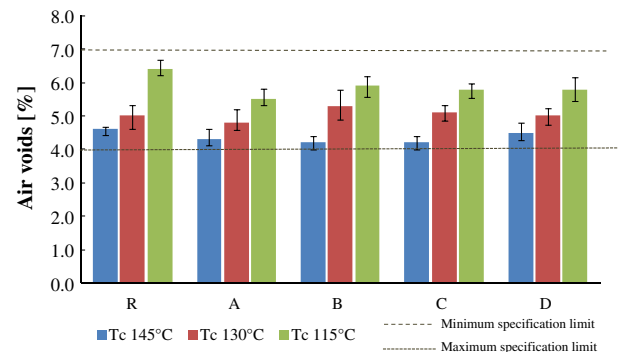


Fig. 5. Air voids obtained in samples from AC 16 W 35/50 with the addition of zeolites.

Indirect Tensile Strength of samples unconditioned (ITS_D) with zeolite addition was always higher than of reference mix asphalt samples with the same compaction temperature (ITS_D from 3.7% to 13%). In the case of the average strength for the conditioned samples (ITS_W) this relationship was observed only for mix asphalt with the addition of unmodified zeolites. The water and frost resistance, according to Polish technical requirements, was maintained for all types of mix asphalt, in each compaction temperature apart from excepting mix asphalt with addition of synthetic zeolite NaP1 at $T = 115\text{ }^\circ\text{C}$ ($ITSR = 77\%$). The highest increase of $ITSR$ indicator was for mix asphalt with synthetic zeolite addition, at compaction temperature of $115\text{ }^\circ\text{C}$. This increase was 10% in relation to $T_c = 115\text{ }^\circ\text{C}$ reference mix asphalt and 5% in relation to $T_c = 145\text{ }^\circ\text{C}$ reference mix asphalt. Lower $ITSR$ indicators for mix asphalt with addition of zeolites soaked with water indicated that over the course of mix asphalt production not entire water was released from the zeolite structure. The results of thermal mineral tests from the zeolite group unambiguously indicated that zeolite water release process occurs in temperature interval from 100 to $400\text{ }^\circ\text{C}$ [56]. Water immobilized in zeolite pores had an effect on significant decrease in resistance after freezing–defrosting cycle (Table 4). HMA with zeolites studies conducted in Poland indicated an improvement of $ITSR$ indicator when modified asphalt was used [57]. The addition of hydrated lime also increased the water resistance of the mix asphalt with zeolite addition [58].

No adhesives were used in this study. When using acid aggregates (granodiorite) adhesives are recommended and can have a positive effect on water resistance.

5.4. Study of stiffness modulus

Average stiffness modulus values are presented in Figs. 6, 7 and 8. As can be seen, stiffness modulus' value for WMA with zeolites in test temperature of $23\text{ }^\circ\text{C}$ is higher in relation to reference mix asphalt regardless of the zeolite material type and mixing temperature. The same correlation was obtained in studies performed in Turkey [33]. At lower temperature, the zeolite addition causes stiffness modulus' increase at $T_c = 145\text{ }^\circ\text{C}$. At $T_c = 145\text{ }^\circ\text{C}$, the measured stiffness modulus for mix asphalt with zeolites is compared with the results obtained for reference mix asphalt (Figs. 7 and 8). The best results were obtained after applying 1% w/m of clinoptilolite and 0.4% of soaked NaP1. With these additions, the values of stiffness modulus of samples compacted at $130\text{ }^\circ\text{C}$ were higher than the results for the reference sample compacted at a temperature of $145\text{ }^\circ\text{C}$, regardless of the temperature of the test.

It can be concluded that the stiffness modulus depends on the mix asphalt compaction temperature and the temperature of the test. In the case of WMA with zeolites, the type of used zeolite

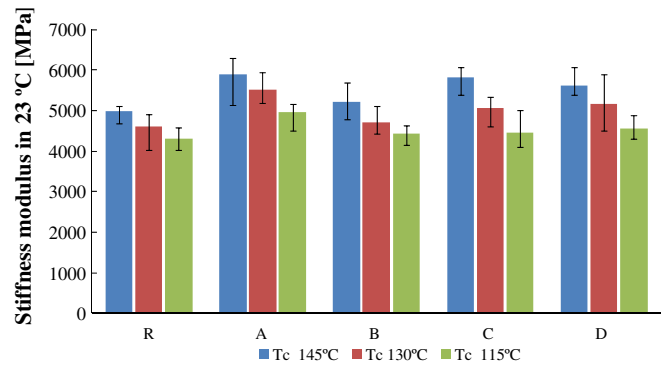


Fig. 6. Average stiffness modulus at 23 °C for the different mix type studied.

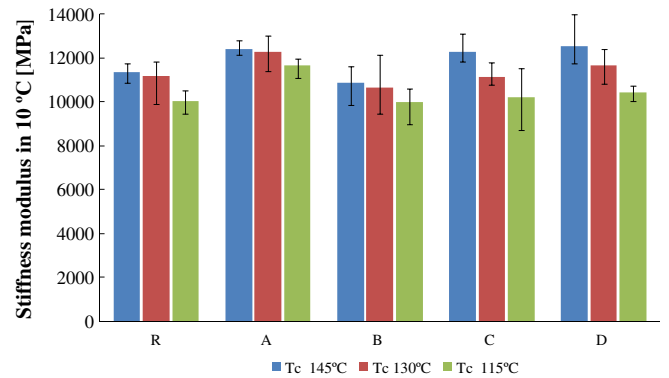


Fig. 7. Average stiffness modulus at 10 °C for the different mix type studied.

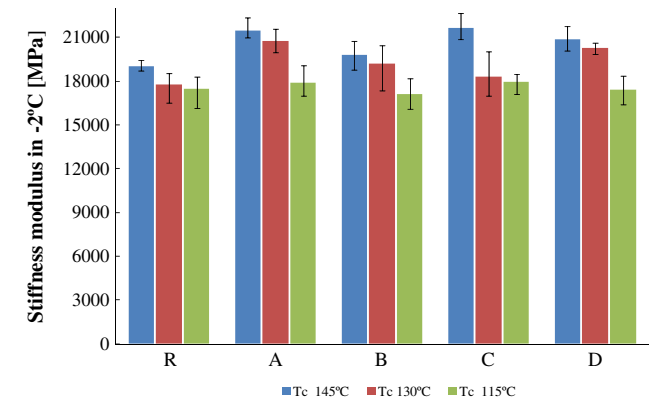


Fig. 8. Average stiffness modulus at -2 °C for the different mix type studied.

Table 4 Results of the indirect tensile strength test for the different mix type studied.

Type of mix	Additive	Amount of additive (%)	Compaction temperature (Tc)											
			145				130				115			
			ITS_W (MPa)	ITS_D (MPa)	$ITSR$ (%)	Air voids (%)	ITS_W (MPa)	ITS_D (MPa)	$ITSR$ (%)	Air voids (%)	ITS_W (MPa)	ITS_D (MPa)	$ITSR$ (%)	Air voids (%)
R	Without additive		0.856	0.928	92	4.6	0.760	0.855	89	5.1	0.721	0.830	87	6.4
A	Natural zeolite clinoptilolite	1	0.951	1.008	94	4.3	0.861	0.990	87	4.9	0.781	0.917	85	5.5
B	Natural zeolite clinoptilolite with water	0.4	0.813	0.941	86	4.2	0.802	0.901	89	5.3	0.713	0.875	81	5.9
C	Synthetic zeolite NaP1	0.5	0.996	1.020	98	4.2	0.820	0.946	87	5.1	0.835	0.861	97	5.8
D	Synthetic zeolite NaP1 with water	0.4	0.858	1.051	82	4.5	0.766	0.950	81	5.0	0.699	0.903	77	5.8

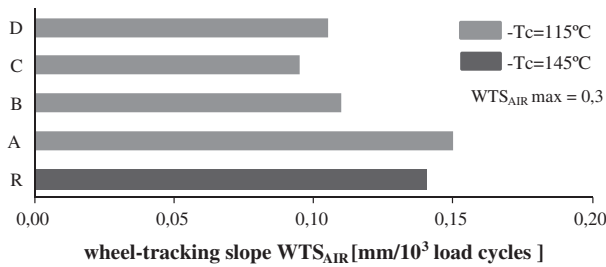


Fig. 9. Wheel-tracking slope for the different mix type studied.

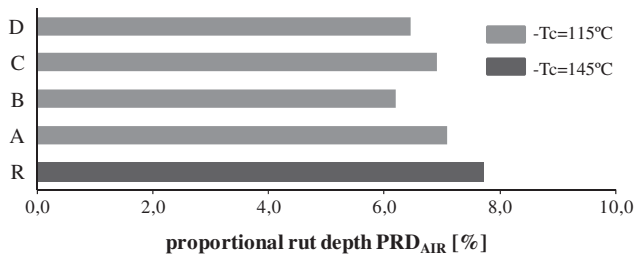


Fig. 10. Proportional rut depth for different mix type studied.

was the main factor responsible for the increase of stiffness modulus in relation to the reference mix asphalt sample.

5.5. Wheel-tracking test

The obtained results of wheel-tracking test are presented in Figs. 9 and 10. Zeolite addition to mix asphalt improved the resistance to wheel-tracking with regard to reference mix asphalt, despite decreasing the compaction temperature by 30 °C. The effect of Na-P1 additive was higher than of clinoptilolite. WMA with zeolite Na-P1 a WTS_{AIR} decreased of 28%. Similar results were obtained when zeolite NaP1 soaked with water was applied. All types of tested mix asphalts, according to Polish technical requirements (WTS_{AIR} MAX = 0.3) were resistant to rutting.

6. Conclusions and recommendations

In the performed tests, physicochemical properties of WMA with addition of different zeolite types (including zeolites soaked with water) were assessed at different compaction temperatures. The decrease of compaction temperature improves the mix asphalt workability, allows to increase the distance and transport time of ready mix asphalt. Also the compaction temperature can be lowered, what in Polish climate conditions may extend the road work season. Car traffic can be returned to a road constructed with this technology in relatively short time. It is particularly significant in case of renovation works in big agglomerations.

Compactability tests in a gyratory compactor indicate a possibility to decrease the compaction temperature through zeolite addition and allow to determine the optimal amount of this additive. The best compactability, measured by the air void content, was obtained for the mix asphalt samples with the following zeolite additives: 1.0% of clinoptilolite; 0.5% of NaP1 and 0.4% of clinoptilolite and NaP1 soaked with water w/m.

Additional soaking of zeolite with water allows to decrease the amount of d natural zeolite from 1% w/m to 0.4% w/m, and the NaP1 synthetic zeolite from 0.5% w/m to 0.4% w/m, but it does not significantly affect the improvement in tested mix asphalt's physicochemical properties. On the other hand, the addition of

zeolites soaked with water causes significant decrease in water and frost resistance, expressed by ITSR indicator.

Synthetic zeolite NaP1 dosing with water, as well as without additional soaking, improves the rut resistance at T_c = 115 °C, in relation to reference mix asphalt with T_c = 145 °C.

After adding the clinoptilolite without soaking to mix asphalt, the higher stiffness modulus were obtained, regardless of the compaction temperature and test temperature.

WMAs with decreased compaction temperature, with zeolite addition meet Polish technical requirements: air void content, ITSR and rut resistance. NaP1-type synthetic zeolite produced from fly ash as well as natural clinoptilolite may constitute an promising alternative to the conventional additives used in mix asphalt technologies.

Acknowledgement

The study was supported by Statutory Fund No.: S-50/II/B/2014 and S12/II/B/2015.

References

- [1] EN 13108-1 (Ed.), Bituminous Mixtures – Material Specifications – Part 1: Asphalt Concrete, 2008.
- [2] M.C. Rubio, G. Martínez, L. Baena, F. Moreno, Warm mix asphalt: an overview, *J. Clean. Prod.* 24 (2012) 76–84.
- [3] A. Vaitkus, D. Cygas, A. Laurinavicius, Z. Perveneckas, Analysis and evaluation of possibilities for the use of warm mix asphalt in Lithuania, *Baltic J. Road Bridge Eng.* 4 (2009) 80–86.
- [4] J. D'Angelo, E. Harm, J. Bartoszek, G. Baumgardner, M. Corrigan, J. Cowsert, Warm-Mix Asphalt: European Practice, Report No. FHWA-PL-08-007, Federal Highway Administration, USA, 2008.
- [5] A. Bernier, A. Zofka, I. Yut, Laboratory evaluation of rutting susceptibility of polymer-modified asphalt mixtures containing recycled pavements, *Constr. Build. Mater.* 31 (2012) 58–66.
- [6] A. Bernier, A. Zofka, R. Josen, J. Mahoney, Warm-mix asphalt pilot project in Connecticut, *Transp. Res. Rec.* 2294 (2012) 106–116.
- [7] M. Zaumanis, Warm Mix Asphalt Investigation (Ph.D. thesis), Riga Technical University, Kgs. Lyngby, Denmark, 2010.
- [8] L.H. Csanyi, Foamed asphalt in bituminous paving mixes, *Highway Res. Board Bull.* 10 (1957) 108–122.
- [9] S. Perkins, Synthesis of warm mix asphalt paving strategies for use in Montana highway construction, in: Montana: the State of Montana: Department of Transportation Final Report, 2009.
- [10] K.J. Jenkins, Mix Design Consideration for Cold and Half-warm Bituminous Mixes with Emphasis on Foamed Bitumen (Ph.D. dissertation), University of Stellenbosch, South Africa, 2000.
- [11] P.J. Ruckel, S.M. Ascott, R.H. Bowering, Foamed-asphalt paving mixtures: preparation of design mixes and treatment of test specimens, *Transp. Res. Board* (1982) 88–95.
- [12] D.Y. Lee, Treating marginal aggregates and soils with foamed asphalt, *Proc. Assoc. Asphalt Paving Technol.* 50 (1981) 211–250.
- [13] L.N. Mohammad, M.Y. Abu-Farsakh, Z. Wu, C. Abadie, Louisiana experience with foamed recycled asphalt pavement base materials, in: Transportation Research Board 82nd Annual Meeting (CD-Rom), 2003. Washington, DC, USA.
- [14] O.R. Larsen, O. Moen, C. Robertus, B.G. Koenders, WAM-Foam asphalt production at lower operating temperatures as an environmental friendly alternative to HMA, in: Proceedings 3rd Eurasphalt & Eurobitume Congress, 2004. Vienna, Austria.
- [15] W. Franus, Characterization of X-type zeolite prepared from coal fly ash, *Pol. J. Environ. Stud.* 21 (2012) 337–343.
- [16] M. Wdowin, W. Franus, R. Panek, Preliminary results of usage possibilities of carbonate and zeolitic sorbents in CO₂ capture, *Fresenius Environ. Bull.* 21 (2012) 3726–3734.
- [17] S. Chałupnik, W. Franus, M. Wysocka, G. Gzyl, Application of zeolites for radium removal from mine water, *Environ. Sci. Pollut. Res.* 20 (2013) 7900–7906.
- [18] W. Franus, K. Dudek, Clay minerals and clinoptilolite of variegated shales formation of the skole unit. Polish flysch carpathians, *Geol. Carpathica* 50 (1999) 23–24.
- [19] W. Franus, M. Wdowin, M. Franus, Synthesis and characterization of zeolites prepared from industrial fly ash, *Environ. Monit. Assess.* 186 (2014) 5721–5729.
- [20] W. Franus, Materiał zeolitowy typu X otrzymany z popiołu lotnego w wyniku syntezy hydrotermalnej i niskotemperaturowej, *Budownictwo i Architektura* 7 (2010) 25–34.
- [21] R. Vaiana, T. Iuele, V. Gallelli, Warm mix asphalt with synthetic zeolite: a laboratory study on mixes workability, *Int. J. Pavement Res. Technol.* 6 (2013) 562–569.

- [22] T. Gandhi, W. Rogers, S. Amirghani, Laboratory evaluation of warm mix asphalt ageing characteristics, *Int. J. Pavement Eng.* 11 (2010) 133–142.
- [23] E. Sanchez-Alonso, A. Vega-Zamánillo, D. Castro-Fresno, M. DelRio-Prat, Evaluation of compactability and mechanical properties of bituminous mixes with warm additives, *Constr. Build. Mater.* 25 (2011) 2304–2311.
- [24] H. Kim, S.-J. Lee, S.N. Amirghani, Rheology of warm mix asphalt binders with aged binders, *Constr. Build. Mater.* 25 (2011) 183–189.
- [25] D. Jones, R. Wu, B.-W. Tsai, Q. Lu, J.T. Harvey, Warm-mix Asphalt Study: Test Track Construction and First-level Analysis of Phase 1 HVS and Laboratory Testing, 2008 (California).
- [26] T. Aschenbrenner, B. Schiebel, R. West, Three-Year Evaluation of the Colorado Department of Transportation's Warm Mix Asphalt Experimental Feature on I-70 at Silverthorne, Colorado Department of Transportation, Colorado, 2011.
- [27] C. Estakhri, J. Button, A.E. Alvarez, Field and Laboratory Investigation of Warm Mix Asphalt in Texas, Texas Transportation Institute, The Texas A&M University, 2010.
- [28] Information materials from Aspha-min company, available at: <<http://www.aspha-min.com/98.html>>.
- [29] M. Tao, R.B. Mallick, Effects of warm-mix asphalt additives on workability and mechanical properties of reclaimed asphalt pavement material, *Transp. Res. Rec.* 2126 (2009) 151–160.
- [30] Z. Hossain, M. Zaman, E.A. O'Rear, D.-H. Chen, Effectiveness of water-bearing and anti-stripping additives in warm mix asphalt technology, *Int. J. Pavement Eng.* 13 (2012) 424–432.
- [31] B. Sengoz, A. Topal, C. Gorkem, Evaluation of natural zeolite as warm mix asphalt additive and its comparison with other warm mix additives, *Constr. Build. Mater.* 43 (2013) 242–252.
- [32] A. Topal, P.A. Dokandari, Laboratory comparison of aging characteristics of warm mix asphalts involving natural and synthetic water containing additives, *Mater. Res.* 17 (2014) 1129–1136.
- [33] A. Topal, B. Sengoz, B.V. Kok, M. Yilmaz, P.A. Dokandari, J. Oner, D. Kaya, Evaluation of mixture characteristics of warm mix asphalt involving natural and synthetic zeolite additives, *Constr. Build. Mater.* 57 (2014) 38–44.
- [34] W. Barthel, J. Marchand, M. Von Devivere, Warm mix asphalt by adding a synthetic zeolite, in: *Proceedings of the Third Eurasphalt and Eurobitume Conference*, Book 1, Foundation Eurasphalt, Breukelen, the Netherlands, 2004, pp. 1241–1249.
- [35] A. Wozzuk, Projektowanie mieszanki mineralno-asfaltowej na ciepło z dodatkiem zeolitu, *Budownictwo i Architektura* 12 (2013) 41–49.
- [36] EN 1426 (Ed.), Bitumen and Bituminous Binders - Determination of Needle Penetration, 2009.
- [37] EN 12593 (Ed.), Bitumen and Bituminous Binders - Determination of the Fraass Breaking Point, 2009.
- [38] EN 1427 (Ed.), Bitumen and Bituminous Binders - Determination of Softening Point - Ring and Ball method, 2009.
- [39] ASTM D 4402 - Standard Test Method for Viscosity Determination of Asphalt at Elevated Temperatures Using a Rotational Viscometer.
- [40] EN 12591 (Ed.), Bitumen and Bituminous Binders - Specifications for Paving Grade Bitumens, 2010.
- [41] EN 933-1 (Ed.), Tests for Geometrical Properties of Aggregates - Part 1: Determination of Particle Size Distribution - Sieving Method, 2012.
- [42] EN 933-10 (Ed.), Tests for Geometrical Properties of Aggregates - Part 10: Assessment of Fines - Grading of Fillers (Air Jet Sieving), 2009.
- [43] Asphalt surfaces on national roads, Technical requirements WT2, 2010.
- [44] L. Bandura, R. Panek, M. Rotko, W. Franus, Synthetic zeolites from fly ash for an effective trapping of BTX in gas stream, *Microporous Mesoporous Mater.* 223 (2016) 1–9.
- [45] L. Bandura, M. Franus, G. Józefaciuk, W. Franus, Synthetic zeolites from fly ash as effective mineral sorbents for land-based petroleum spills cleanup, *Fuel* 147 (2015) 100–107.
- [46] M. Franus, M. Wdowin, L. Bandura, W. Franus, Removal of environmental pollutions using zeolites from fly ash: A review, *Fresenius Environ. Bull.* 3 (2015) 854–866.
- [47] EN-12697-31 (Ed.), Bituminous Mixtures - Test Methods for Hot Mix Asphalt - Part 31: Specimen Preparation by Gyrotory Compactor, 2007.
- [48] EN 12697-30 (Ed.), Bituminous Mixtures - Test Methods for Hot Mix Asphalt - Part 30: Specimen Preparation by Impact Compactor, 2012.
- [49] EN 12697-8 (Ed.), Bituminous Mixtures - Test Methods for Hot Mix Asphalt - Part 8: Determination of Void Characteristics of Bituminous Specimens, 2005.
- [50] PN-EN 12697-12 (Ed.), Bituminous Mixtures - Test Methods for Hot Mix Asphalt - Part 12: Determination of the Water Sensitivity of Bituminous Specimens, 2008.
- [51] EN 12697-23 (Ed.), Bituminous Mixtures - Test Methods for Hot Mix Asphalt - Part 23: Determination of the Indirect Tensile Strength of Bituminous Specimens, 2009.
- [52] EN 12697-26 (Ed.), Bituminous Mixtures - Test Methods for Hot Mix Asphalt - Part 26: Stiffness, 2012.
- [53] Minister's of Transport and Maritime Economy Regulation of 2nd March 1999 regarding the technical conditions which should be met by public roads and their locations, (*Dz. U. Journal of Laws*, 1999 No 43 pos. 430).
- [54] EN 12697-22+A1 (Ed.), Bituminous Mixtures - Test Methods for Hot Mix Asphalt - Part 22: Wheel Cracking, 2008.
- [55] EN 12697-33+A1 (Ed.), Bituminous Mixtures - Test Methods for Hot Mix Asphalt - Part 33: Specimen Preparation, Slab Compactom, 2008.
- [56] A. Grela, T. Bajda, J. Mikula, The mineral composition and textural properties of zeolites with metakaolin, *Przemysł Chemiczny* 94 (4) (2015) 619–622.
- [57] J. Judycki, Badania mieszanek mineralno-asfaltowych o obniżonej temperaturze otaczania, Projekt badawczy dla GDDKiA, Gdańsk, 2011.
- [58] G.C. Hurley, B.D. Prowell, Evaluation of Potential Processes for Use in Warm Mix Asphalt, National Center for Asphalt Technology, Auburn University, Alabama, 2006.