Mechanical Behavior of Asphalt Mixtures Containing Silica Gels as Warm Additives

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Abstract

This paper presents the results of a study of some compounds capable of absorbing water into their structure (silica gel), as potential foaming binders. Asphalt mixtures were manufactured at different manufacturing and compaction temperatures, using four different silica gels. Static and dynamic tests were carried out to determine their behavior in asphalt mixtures. The results were compared with those obtained using hot-mix asphalt and warmmix asphalt manufactured with zeolite. The lab results showed a similar behavior of asphalt mixtures containing either silica gel or zeolite.

Keywords: silica gel; zeolite; additives; warm asphalt mixtures; rutting; fatigue

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1. Introduction

Different technologies are used to reduce energy consumption during manufacture and compaction of asphalt mixtures, some based on the use of additives and others on the use of water. These water technologies can be used with a system of nozzles (e.g. Double Barrel®), wet aggregates during the production process (e.g. WAM Foam® or LEA®) in order to produce foaming binders or additives can be used within the mix to achieve a foam of bitumen (e.g. zeolites) [1]. These methods achieve a reduction of the binder viscosity, coating the aggregates at lower temperatures.

Zeolites are aluminosilicate compounds, and depending on the type of zeolite have a pore size from 3 Å to 8 Å. Due to this porous structure, they have an ion exchange capacity and can contain different molecules [2]. The zeolites used in the asphalt industry contain crystallisation water in a percentage close to 20% by weight. Adding zeolites during the manufacture of asphalt mixtures, their water content is released foaming the bitumen, obtaining an improvement in the coating of aggregates and the manageability of the asphalt mixture at lower temperatures [3].

Several authors have studied the behavior of warm-mix asphalts (WMA) with zeolites (Aspha-Min®), finding that their use improved the mixture compaction, the resilient modulus was not affected and did not increase the susceptibility to rutting of asphalt mixtures [4]. Akisetty et al. [5] concluded that it is possible to reduce the compaction temperatures in crumb rubber-modified asphalt mixtures using additives, among them the zeolite Aspha-Min®, achieving a suitable percentage of air voids. Tao and Mallick [6] showed that Reclaimed Asphalt Pavement (RAP) content could be increased to 100% through the use of the zeolite Advera®WMA, obtaining an increase in stiffness of the asphalt mixture at temperatures below 80°C and enhancing the indirect tensile strength.

compaction, as well as the rheological properties to check the performance of asphalt mixtures manufactured with zeolite, an organic additive and a chemical additive. According to the results obtained, the mixing and compaction temperatures are reduced between 6-13°C and 6-10°C, respectively; depending on the type of additives used. The optimal additive content is 3% w/b for the organic and chemical additives and between 5-6% w/b for zeolites for use in heavy traffic and high temperature conditions.

Xu et al. [8] made a review about the moisture damage behavior of WMA evaluating the effects of different types of technologies, type of aggregate, type of binder and use of additives as influential factors in resistance to moisture damage of WMA. Among the conclusions, the authors indicated that in foaming technologies it is crucial to verify the optimum percentage of additive, and optimize the mixing temperature. Both factors will significantly influence in achieving a better coating of aggregates and decrease the water content trapped in the asphalt mixture, influential factors in the resistance to the moisture damage.

De Visscher et al. [9] studied the mechanical properties of asphalt mixtures using different zeolites. The results indicated that reducing the compaction temperature below 120°C, the use of zeolites had a negative effect on the compactability and water sensitivity; but reducing the manufacture temperature 30°C did not lead to a negative effect on the rutting resistance compared to a reference mixture manufactured at 150°C. Woszuk and Franus [10] evaluated the mechanical properties of synthetic and natural zeolites, which have 28% and 17% water in their structure, respectively. Three compaction temperatures in the design of the mixtures (145°C, 130°C and 115°C) were used; obtaining an optimum content of natural zeolite of 1% w/m and 0.5% w/m for the synthetic one. The results of water sensitivity and permanent deformations showed a good performance with both zeolites according to Polish standards.

Hossain et al. [11] demonstrated that the addition of 6% by weight of bitumen of the zeolite Advera®WMA did not change the PG grade of the original binder. They also aged the bitumen using the rolling thin film oven test (RTFOT) at a temperature of 150°C and tested with dynamic shear rheometer (DSR), reducing the rutting resistance of the modified binder, although the authors indicated that it could be due to the reduction of manufacture temperature rather than the use of the additive. Topal et al. [12] evaluated the Marshall stability, stiffness modulus and indirect tensile fatigue of asphalt mixtures manufactured with natural and synthetic zeolites. According to the data shown by the authors, there would be a slight decrease in the amount of binder needed with respect to the conventional hot asphalt mixture, which it would mean an initial cost savings. The use of both zeolites increased the stiffness of the asphalt mixture. However, the results of fatigue resistance and permanent deformation were slightly better with the synthetic zeolite.

Wu and Li [13] studied the curing time for WMA manufactured with synthetic zeolite (Advera®WMA), and tested their behavior in dynamic modulus, rutting, resistance to moisture damage and fatigue behavior. The results showed that the WMA properties improved with the curing time, probably due to the elimination of water contained in the asphalt mixture, unlike hot asphalt mixture which has not shown difference in its performance after the curing time evaluated.

Goh and You [14] studied dynamic moduli and four-point beam fatigue of asphalt mixtures with wax (Sasobit®) and zeolite (Advera®WMA) additives, manufactured and compacted at different temperatures. The dynamic moduli of WMA were lower than hot mix asphalt (HMA) at most temperatures and frequencies tested and, the greater the compaction temperature of WMA, the greater the modulus value. The mixtures with zeolites were less stiff than wax mixtures. WMA showed similar or higher fatigue life than HMA, except when they were manufactured and compacted at 130°C.

Silica gels are amorphous silicon dioxides, very porous, with high surface area [15]. Due to their porosity and surface area, the silica gels could be used to separate and adsorb molecules, in high performance liquid chromatography, in membrane technology and molecular engineering, in chemical-mechanical surface treatment, among other [16]. The pore size is usually between 5 Å and 300 Å, depending on the synthesis process used [16]. The adsorption capacity of silica gel is based on physical adsorption, that is, the molecules are bound by Van der Waals forces at the surface [17]. The regeneration of silica gels, releasing molecules adsorbed, could be carried out by three methods: increasing the temperature, lowering the molar concentration of the adsorbate or lowering of the system pressure [17]. The regeneration at high temperatures can be carried out by heating up to 800°C depending on the absorbed components, being in the case of water molecules above 100°C [18].

Due to the water absorption-desorption capacity of the silica gels, and the release of water at the manufacture temperature of WMA without affecting the structure of the silica gel whose melting point is higher than 1600°C [19]; these materials may be an alternative to the use of synthetic zeolites for the manufacture of WMA.

Therefore, this paper evaluate if silica gels may be used as alternative additives in WMA. The foamed asphalt mixture has been simulated using several silica gels, comparing their results with WMA manufactured with zeolite. The ease of compaction and static and dynamic mechanical properties have been studied in asphalt mixtures modified with some additives (zeolite and silica gels) at different manufacturing and compaction temperatures, also comparing their results with HMA.

2. Experimental Research

This paper studied the mechanical properties of asphalt mixtures containing silica gels, manufactured at different temperatures, to determine whether they may be used as foamed additives.

2.1 Materials used and Mix design

In this paper, an aggregate gradation with maximum aggregate size of 16mm and for use in the surface layer (AC16Surf) was chosen. The AC16Surf aggregate gradation was adjusted to the centre of the grading envelope (Table 1), using ophite coarse aggregates and limestone sand. The aggregates' properties are described in Table 2.

The asphalt mixtures were manufactured with a B-50/70 penetration bitumen (Table 3), using the optimum binder content of 4.5% weight of mix (w/m) obtained with the Marshall procedure [20] and the analysis of air void content [21].

In this research, five additives (F1-F5) were studied, whose characteristics are described in Table 4.

F1 is usually used in WMA and 0.3% w/m was added in as recommended by the manufacturer. The amount of each silica gel additive used (F2-F5) was calculated to add the same amount of water as synthetic zeolite to simulate the foaming effect. For this, the water absorption was calculated in situ as follows: an amount of each silica gel was weighted in a vessel and then distilled water was added until no more water was absorbed, and the silica gel particles were not clustered. Thus, the amount of each silica gel to simulate the foaming process was: 0.06% w/m for F2; 0.02% w/m for F3 and F5, and 0.10% w/m for F4. The five additives were added into the asphalt mixture (it is necessary to hydrate the silica gels before adding them) at the same time as the bitumen to achieve the foaming effect.

The asphalt mixtures were manufactured and compacted at three temperatures: 160°C, typical temperature of HMA; 140°C and 120°C, temperatures of WMA. To maintain the selected manufacture temperature, a mixer with control of temperature was used. This mixer has a mixing bowl with 201 of capacity, thermally insulated.

2.2 Test methods

2.2.1 Adsorption isotherms

The water adsorption isotherms enable to know the amount of balance water of a compound exposed to a temperature and relative moisture known [23], and the relationship between adsorption and porosity of materials [24]. The IUPAC conventions have been proposed six types of isotherms, shown in Fig. 1. Type I isotherm is typical in microporous materials, type II, III and VI isotherms are in nonporous or macroporous materials; and IV and V type isotherms in mesoporous adsorbents [24].

Prior to testing, the samples were pre-dried. The silica gel samples were dried at 110°C in an oven for 20h and the zeolite was kept under vacuum at 200°C, for the same time. Quantachrome FloVac Degasser was used for the vacuum treatment.

The adsorption-desorption water isotherms at 25°C were obtained with the Quantachrome Hydrosorb Model HS-10. The relative moisture inside the machine was increased from 5% to 95% in intervals of 10%. The criteria selected to increase the moisture was that the pressure did not vary more than 0.05 mmHg for one minute, being 10 min the equilibration time chosen. Thus, it ensures that the samples acquire a constant weight for this relative moisture. To calculate the desorption isotherm, the same approach was applied but gradually decreasing the relative moisture.

The ease of compaction of different asphalt mixtures was studied using a gyratory compactor. This ease was measured using the Mix Stability Index (MSI). This index is based on the % air voids-number of cycles curve and on the use of variable limits depending on the aggregate gradation selected. These limits are related to the % air voids obtained with the Marshall test, the method used in Spain to obtain the optimum binder content [25, 26]. MSI was calculated as the area from cycle 8 until achieving 98% of the Marshall density [26]. The compaction parameters applied were: an internal slight angle of 0.82°; a speed of 30 rpm; a vertical pressure of 600 kPa [27] and 200 cycles [28].

2.2.3 Water sensitivity

The binder-aggregate adhesion was carried out through the water sensitivity test [29]. Six cylindrical specimens (diameter 101.6 mm and height 63.5 mm) were manufactured for each combination of variables studied, separating them into two groups with similar densities: one in dry conditions (air chamber at 20°C) and the other in wet conditions (submerged in water at 40°C) after a vacuum treatment. All the specimens were tested for indirect tensile strength [30], calculating the Indirect Tensile Strength Ratio (% ITSR) through the strength results of wet (ITS_w) and dry (ITS_D) specimens.

2.2.4 Stiffness modulus

The stiffness modulus was calculated according to the European Standard UNE EN 12697-26 [31], annex C, using the Indiret Tensile test. A vertical compression load was applied, measuring the horizontal deformation of the cylindrical sample. Each test consists of 16 load cycles of 3 s, using the values from cycle 11 to 15 for the calculation of the stiffness moduli, as the first cycles are necessary for sample settling. The test temperature was 20°C.

2.2.5 Resistance to permanent deformation

The wheel tracking test was used to analyze the permanent deformation of the asphalt mixtures manufactured and compacted at three different temperatures. Two specimens were manufactured for each combination of variables. For an AC16 gradation, the specimens had the dimensions of 260 mm x 410 mm x 60 mm according to UNE EN 12697-22 [32], and they were compacted by a steel segmented roller compactor [33].

The test temperature was 60°C [25] and the test is set to endure either 10,000 loading cycles or until the rut depth is 20 mm [32].

The permanent deformation is measured through mean wheel tracking slope calculated by the following equation:

$$WTS_{air} = \frac{(d_{10,000} - d_{5,000})}{5}$$
 Eq. 1

where:

WTS_{air}: wheel tracking slope for 1,000 loading cycles (mm) $d_{5,000}$: rut depth after 5,000 loading cycles (mm)

d_{10,000}: rut depth after 10,000 loading cycles (mm)

2.2.6 Resistance to fatigue

The fatigue test is used to evaluate the structural behavior of asphalt mixtures. It measures the distress of the asphalt mixtures showed in cracking form, produced by the accumulation of high number of loads [34].

There are many procedures for the fatigue test depending on the geometry of the specimens [35]. In this research, the four-point bending test was used. The AC16 specimens had a width of 63 mm, a length of 410 mm and a thickness of 50 mm according to European

standard UNE EN 12697-24 [35]. The test temperature was 20°C and a sinusoidal waveform at a frequency of 10 Hz was applied in strain control mode. The failure criteria selected is defined as the loading cycle when a reduction of 50% of the initial stiffness modulus is produced.

The fatigue model for parallelepiped specimens tested in the four-point bending test, has the following expression:

$$N = k_1 \varepsilon^{k_2}$$
 Eq. 2

Where:

N: number of strain applications to failure

 ϵ : initial tensile strain in the cycle 100, in μ m/m

k₁, k₂: fatigue growth rate coefficients

3. Results and discussion

3.1 Adsorption isotherms of zeolite and silica gels.

The adsorption-desorption water isotherms at 25°C are shown in Fig. 2. The results indicate that the additives used have different adsorption methods.

The zeolite (F1) showed a type I hybrid isotherm, which indicated that F1 is a microporous compound and the adsorption is produced at lower pressures and with a monolayer mechanism [36]; also showing a hysteresis loop at medium-high pressures, similar to isotherm type IV, due to the presence of mesopores, resulting in capillary condensation in the adsorbent [37, 38]. Thus, F1 has micropores and mesopores.

In the case of silica gels, F2, F3 and F5 presented a type V isotherm, typical in porous compounds with a weak interaction between the adsorbate and the adsorbent, where the adsorption has a multilayer mechanism [36]. In turn, F4 showed a type IV isotherm, typical

of mesoporous solids, whose filling process is in multilayer formation. The difference in the hysteresis loops between silica gels may be due to the shape of the pores, their structures and also the gas used in the test [39].

3.2 Compactability

According to the results shown in Fig. 3, the use of silica gels improved the compactability of asphalt mixtures, probably due to the reduction of binder viscosity. The difference in the compactability of silica gels may be due to their different internal structure, the intermolecular forces generate between adsorbent and adsorbate (whether it is in monolayer or multilayer formations), temperature at which all water content is released, and /or time to release the water content during the manufacture of asphalt mixtures.

Additives F1, F3, F4 and F5 needed lower compaction energies, between 25-55%, to achieve 98% of Marshall density at the three temperatures studied than reference mixture at 160°C (Rmix). The MSI value of additive F2 at 120°C was lower compared to Rmix, being more easily compactable at this temperature.

So, the use of silica gels may reduce the energy consumption of asphalt mixtures due to the lower manufacturing temperatures used, and the number of cycles of the vibratory roller for their layout.

3.3 Analysis of mechanical properties

The water sensitivity test results are shown in Fig. 4. A decrease of % ITSR values has been obtained reducing the working temperatures, due to the increase in air void content.

Adding additives improved the water sensitivity compared to Rmix at the same temperature, although none of them achieved the minimum values of % ITSR indicated in the Spanish standard (>80%) in asphalt mixtures manufactured at 140°C and 120°C. It is remarkable that mixtures with additives manufactured at 140°C obtained % ITSRs close to 80%, and

modifying the dosage of the asphalt mixture and/or the type of bitumen may be achieves this value.

At 160°C, wet and dry tensile strength values of majority of asphalt mixtures with additives, were slightly lower than Rmix. However, at 140°C and 120°C, asphalt mixtures with additives showed greater tensile strength than Rmix, in both dry and wet specimens. These greater resistances may be due to the lower percentage of air voids obtained in modified asphalt mixtures, because all additives used in this research did not melt at the manufacturing temperatures used, behaving them as filler.

In Fig. 5, the average of stiffness modulus of four specimens tested for each temperatureadditive combination is shown. Reducing the manufacture temperature, the stiffness moduli decreased in all asphalt mixtures tested because of the increase in air void content. A decrease of 40°C in the manufacture and compaction temperatures decreased the moduli of asphalt mixtures with additives between 10-35% depending on the additive used, but in Rmix this reduction was around 50%.

The results of rutting performance tested at 60°C are shown in Table 5. In all cases studied, the permanent deformation increased when the manufacturing and compaction temperatures decreased for the same asphalt mixture. This effect is due to the higher percentage of air voids obtained when reducing the work temperatures, causing a lower rutting resistance. However, comparing the asphalt mixtures at the same manufacture temperature, the percentage of air voids was not the only reason of their rutting behavior, because additivated asphalt mixtures with the same air void content did not obtain the same results. Analysing the rutting results taking into account the characteristics of the additives shown in Table 4, the surface area, particle size or silica content have not shown a clear relationship with the rutting results. Nevertheless, there seems to be a relationship between the pore volume of the different silica gels and the resistance to rutting and, the greater pore volume of additive, the

greater the WTS results. In the case of F4, whose WTS and RD values always turned out to be the greatest of all the asphalt mixtures evaluated, it may also be due to a specific characteristic of this additive (Table 4): the release of heat by the adsorption of substances, softening the bitumen of the asphalt mixture at the test temperature. All asphalt mixtures with additives showed higher rut depth than Rmix at the same working temperature. Among them, additives F2 and F1 obtained lower permanent deformation than the rest of additives at three manufacturing and compacting temperatures.

The fatigue resistance was analyzed for an HMA and two additives (F1 and F2), which obtained better results in terms of permanent deformation. In this case, the asphalt mixture with additives was manufactured and compacted at 140°C (temperature of WMA).

The asphalt mixtures with additives had a higher slope in plots of fatigue tests than HMA (Table 6), which indicates that these asphalt mixtures are more susceptible to change of strain level [40]. Comparing the value obtained at 10^6 cycles, the use of both additives resulted in a lower fatigue life, 7% in the case of F1 and 15% to F2 compared to HMA.

4. Conclusions

In this paper, the use of silica gels has been studied as possible foaming agents for bitumen. Asphalt mixtures manufactured with silica gels showed similar behavior to asphalt mixtures with zeolite, being F2 the silica gel that achieved better performance. The use of silica gels improved the workability of the asphalt mixtures compared to HMA. F3, F4 and F5 obtained lower MSI values (between 25% and 55%) to HMA values.

High tensile strength values were obtained for asphalt mixtures with additives compared to Rmix at the same manufacturing and compaction temperature, obtaining an improvement in % ITSR. Although none of the additivated mixtures manufactured at lower temperatures

achieved the %ITSR indicated in the Spanish standard, modifying the dosage of the asphalt mixture and/or the type of bitumen may be achieved it.

Decreasing the manufacture temperature, the asphalt mixtures became less stiff, but with the use of silica gels and zeolite, this reduction of stiffness with temperature was lower than in Rmix. All additives used showed poorer permanent deformation resistance than Rmix at the three temperatures studied. Among them, additives F1 and F2 have better performance. Asphalt mixtures manufactured with F1 and F2 at 140°C obtained a lower fatigue life, between 7-15%, than HMA.

It is recommended to carry out a research varying the binder penetration and the type of aggregate, as well as the possible use of recycled materials (e.g. RAP) to improve the results and evaluate if it is possible to fulfill the standard requirements. It is also recommended to carry out a research to know the water release temperature of each silica gel studied as well as the time required to release all water adsorbed in silica gels during the manufacture of asphalt mixtures.

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Conflicts of Interest

The authors declare no conflict of interest.

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Figure captions

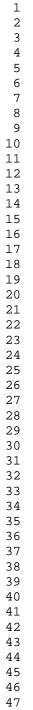
Fig. 1. IUPAC classification adsorption-desorption isotherms [24].

Fig. 2. Adsorption-desorption water isotherms at 25°C.

Fig. 3. Evaluation of compactability.

Fig. 4. Tensile strength of different asphalt mixtures.

Fig. 5. Stiffness moduli at 20°C.





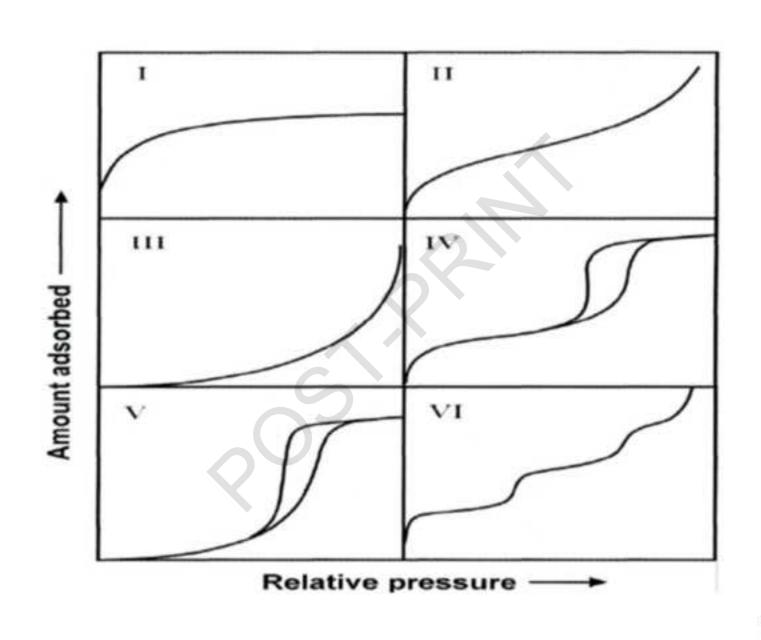


Fig. 1





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4,000 3,500 3,000 2,500 MSI 2,000 1,500 1,000 120°C 160°C 140°C 140°C 140°C 120°C 140°C 160°C 140°C 120°C 160°C 120°C 160°C 140°C 120°C 160°C 160°C

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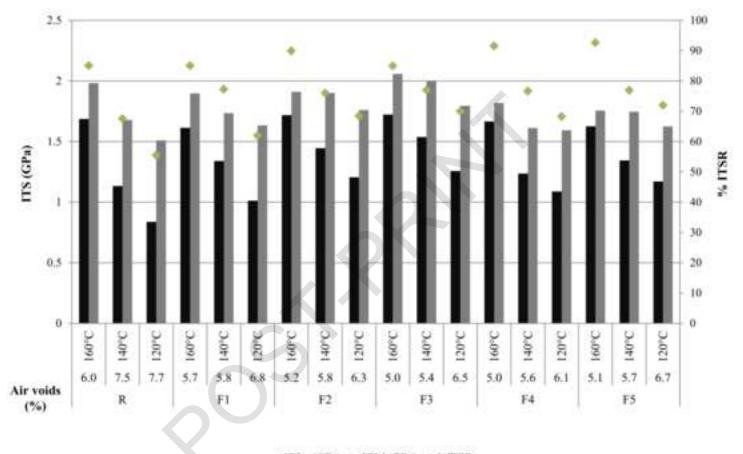
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Fig. 3

120°C

F5

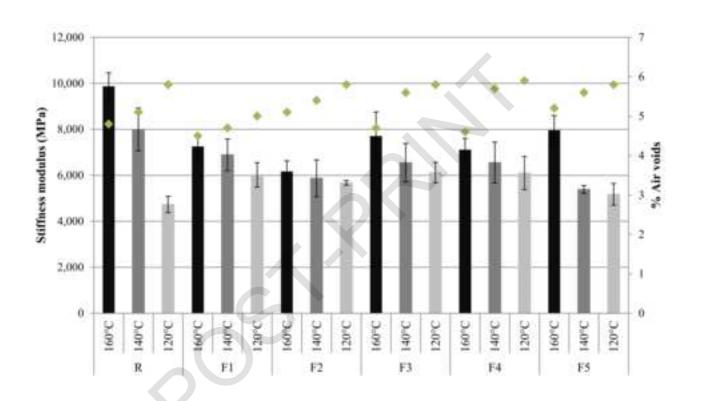
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■ITSw (GPa) ■ITSd (GPa) ◆%ITSR

- 49

Fig.4



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Fig. 5

Particle size, D (mm)		22	16	8	4	2	0.500	0.250	0.063
	Max.	100	100	75	50	38	21	15	7
Percent passing (%)	Min.	100	90	60	35	24	11	7	3
	Selected gradation	100	95	67.5	42.5	31	16	11	5

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 Table 1. AC16Surf aggregate gradation.

Table 2. Aggregate properties.

Properties	Standard method	Criteria	Test results
Bulk specific gravity (kg/m ³)	EN 1097-6	n/a	2859
LA abrasion (%)	EN 1097-2	<20	13
Flakiness index (%)	EN 933-3	<25	12
Sand equivalent (%)	EN 933-8	>50	60

Table 3. Binder - physical properties.

Test	Standard method	Criteria	Test results
Penetration (dmm)	EN 1426	50-70	55
Specific gravity at 25°C (kg/m ³)	EN 15326	>1.0	1031
Ductility at 25°C (cm)	EN 13589	>90	>90
Softening point (°C)	EN 1427	46-54	>48
Fraas breaking point (°C)	ISO 12593	≤-8	<-8
Flash point (°C)	ISO 2592	≥230	>235
Penetration of residue, % of original		>50	>50
	6		

Terminology	Chemical composition	State of additive	Regular use	Characteristics	Particle size (µm)	Pore volume (cc/g)	Surface area (m²/g)
F1	Synthetic zeolite (21% in mass of water), sodium aluminosilicate [Na ₂ O:χSiO ₂ :Al ₂ O ₃ :γH ₂ O]*	Powder form	WMA	Improve the manageability of the mix and the laying process	350	0.18	3.1
F2	Synthetic amorphous silica [99.95% SiO ₂]	Powder form	Chromatography	Separation of compounds by molecular size.	63	0.727	496
F3	Synthetic amorphous silica [98% SiO ₂]	Flowing powder	Anti-caking and industrial applications	High surface area and absorption capacity	16	1.2	190
F4	Synthetic amorphous silica [95-100% SiO ₂]	Granular	Refrigerant drying solid cores, in desiccators and as desiccant in insulating glass units	High water capacity. In active conditions, it will release heat when adsorbing water or other substances.	30	2.1	500
F5	Synthetic amorphous silica [99.4% SiO ₂]	Flowing Powder	Matting agent in thin film coating systems and free flow additive	High porosity, fine particle size and high affinity for moisture	6.5	1.5	300
[*] General formul	a for zeolites. χ and γ values depend or	n the structure ty	pe of zeolite [22]				
			4				

Table 4. Additives used in this research.

	Manufacturing Temperature								
	160 °C			140°C			120°C		
Mix	WTS _{air} (mm/10 ³ cycles)*	RD _{air} (mm) ^{**}	Air voids (%)	WTS _{air} (mm/10 ³ cycles)	RD _{air} (mm) ^{**}	Air voids (%)	WTS _{air} (mm/10 ³ cycles)	RD _{air} (mm)**	Air voids (%)
R	0.06	2.72	5.0	0.10	3.47	5.6	0.14	3.61	6.3
F1	0.12	3.45	4.7	0.15	3.82	5.2	0.19	4.32	5.6
F2	0.10	3.16	4.8	0.14	3.38	5.8	0.16	3.97	6.2
F3	0.15	4.23	3.9	0.17	4.48	4.3	0.21	5.61	5.1
F4	0.16	4.37	4.1	0.20	5.84	4,3	0.30	6.71	5.2
F5	0.15	3.90	3.8	0.17	4.82	4.4	0.21	4.98	4.7

Table 5. Resistance to rutting at 60°C of AC16Surf B-50/70.

*WTSair: mean wheel tracking slope; **RDair: mean rut depth.

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Mix	Manufacture Temperature (°C)	Fatigue Line	R ²	Fatigue Resistance, ε ₆ (μm/m)
R	160	$N=10^{22.184} (\epsilon)^{-6.679}$	0.946	208.5
F1	140	$N=10^{20.924} (\epsilon)^{-6,531}$	0.978	192.9
F2	140	$N=10^{17.699}(\epsilon)^{-5.206}$	0.985	176.7

Table 6. Results of four-point bending fatigue test.

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