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Visual Abstract

Mechanical performance of intelligent asphalt mixture utilizing rejuvenator encapsulated method

Sari Sharif Ali, Mahyar Arabani
Dept. of Civil Engineering, University of Guilan, Rasht, Iran
sarisharif@yahoo.com, m_arbani@yahoo.com

Hassan Latifi
Department of Civil Engineering, Science and Research Branch, Islamic Azad University, Tehran, Iran
Hassan.latifi@srbiau.ac.ir

ABSTRACT. In this study, in order to evaluate the effects of heavy vacuum slops (H.V.S) as rejuvenator and nano-Zycosil as an anti-stripping agent on used encapsulation method and mechanical performance of asphalt mixture samples, scanning electron microscopy (SEM), computerized tomography (CT) scan and thermal gravimetric (TG) analyses and also, indirect tensile strength (ITS) and indirect tensile fatigue (ITF) tests were performed. First, an encapsulation procedure to prepare different specimens including modified and unmodified samples with nano-Zycosil was done. In the following, morphology of the nano-Zycosil-modified capsules and aggregates were particularly evaluated. Considering the morphology evaluation and TG analysis diagrams, it was found that most of the capsules resisted the mixing procedure of the asphalt mixture. So, the encapsulation procedure used in this study was a successful technique. In addition, modification with nano-Zycosil as an anti-stripping agent significantly improved the adhesion strength in the matrix of capsules-aggregates-asphalt binder by converting the adhesion type from silanolian to siloxane. Overall, modification of capsules and main aggregates together with nano-Zycosil significantly improved moisture resistance and mechanical performance of asphalt mixture samples.

KEYWORDS. Rejuvenator capsule; Nano-Zycosil; Indirect tensile; Encapsulation procedure; Moisture resistance.

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INTRODUCTION

Asphalt mixture is a porous material consists of asphalt binder, aggregates and air voids formed at high temperatures (~180 °C) during mixing process [1]. In long term aging during service life, the stiffness of asphalt mixture increases which results in an increase of asphalt binder's brittleness due to oxidation and consequently development of micro-cracks between aggregates and asphalt binder [2,3]. In addition, this procedure also occurs due to being exposd to heat, oxygen and ultraviolet (UV) light during storage, mixing, transport and laying down of asphalt mixture [4,5,6]. Asphalt binder has usually two major components namely: asphaltenes and maltenes which are solid and liquid, respectively. The components of maltenes are polar aromatics (PAs) and naphthalene aromatics (NAs) [7]. Liu et al. [8] found that asphalt binder produced asphaltenes and PAs during aging process. In other words, PAs and NAs transformed into asphaltenes and PAs, respectively. Afterwards, the produced PAs transformed into asphaltenes too. Finally, the asphaltenes component increased. On the other hand, PAs and NAs decreased. In other words, the solid and liquid components increased and decreased, respectively. As a result, the brittleness of asphalt mixture increased [9].

One of the popular methods in order to restore the asphalt binder's properties is using of rejuvenators. Rejuvinating agents consist of maltnes of lubricating oils, have a important goal of restoring asphaltenes/maltenes ratio [10]. On the other hand, successful mixing of rejuvinating agents with asphalt binder and aggregates is very important due to the problem of penetration of them to pavement surface during paving operation. Shen et al in [10] reports the same difficulties by using rejuvinators as they did not penetrate into asphalt binder more than 2 cm. Also, reduction in surface friction of the pavement and the environmental contaminations were the other concers. On these matters, some researches let to encapsulate rejuvenators in order to use in asphalt mixtures [11]. In order to make capsules of rejuvenators, porous sand and epoxy resin-cement were chosen to cover the rejuvenators like a wall. One of the advantages of these capsules is their high resistance at high temperatures during mixing process. In addition, they could remain unbroken until the rejuvenators are necessary in resoring asphalt binder properties. On the other hand, reduction in mechanical resistance of the asphalt mixture is one of disadvantages of the encapsulation method. Moreover, during the mixing process, a little amount of capsules would break which results in releasing the rejuvenators into asphalt binder and soften it. Considering, capsule surface is smoother than the aggregate, so, the adhesive resistance between the asphalt binder and capsules is less than the adhesive resistance between the asphalt binder and aggregates and also, modification of asphalt binder with the capsules was by mass replacement of aggregates. So, indirect tensile and indirect tensile fatigue strengths of the asphalt mixture samples decreased [12,13]. In this study, in order to evaluate the effects of used rejuvenator encapsulated method and nano-Zycosil as an anti-stripping agent on mechanical performance of asphalt mixture samples, scanning electron microscopy (SEM), computerized tomography (CT) scan and thermal gravimetric (TG) analyses and also, indirect tensile strength (ITS) and indirect tensile fatigue (ITF) tests were performed. All the analyses and tests were conducted in Erbil asphalt laboratory in Iraqi Kurdistan. Finally, a chart of the subjects of this study is shown in Fig. 1.

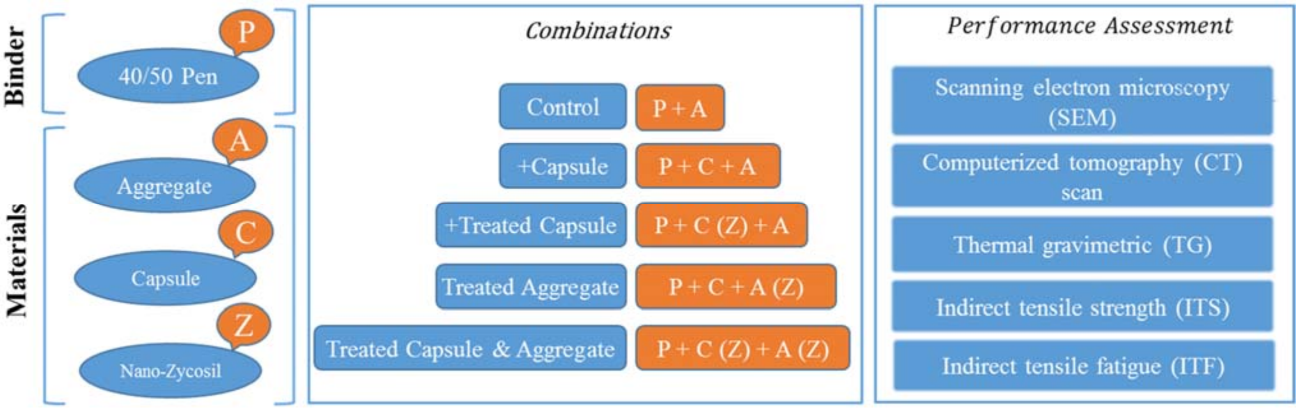


Figure 1: Chart of subjects of this study.

EXPERIMENTAL PROCEDURE

Materials

Main materials used in the encapsulation included scoria porous sand. The particles sizes were between 3 and 4 mm. This type of sand has thousands of micro pores and simply absorbs liquids. This size of porous sand was selected by mass replacement of aggregates in asphalt mixtures. The used rejuvenator is heavy vacuum slops

(H.V.S) which is very dense and aromatic, obtained from Baiji oil refinery in Iraq. The heavy vacuum slops (H.V.S) is purchased from Erbil asphalt laboratory in Iraqi Kurdistan. The wall of capsules is formed of cement Type 1 bonded with a liquid epoxy resin (Bisphenol A). Properties of the materials forming the capsules are illustrated in Tab. 1.

Properties	Density	Specific weight	Porosity
Unit	g/cm^3	g/cm^3	-
Rejuvenator	0.926	9.4	-
Porous sand	2.58	1.54	49.08
Cement	3.1	63.9	-
Epoxy	1.13	1.14	-

Table 1: Properties of the materials forming the capsules.

In the common asphalt mixture samples, crushed aggregates alongside sand and Type 1 cement as filler were used according to Iraqi standard specifications for road and bridge (ISSRB) and it is displayed in Tab. 2. Also, as it is shown in Tab. 3, the used asphalt binder was 40/50-penetration grade from Baiji oil refinery.

Sieve (mm)	19	12.5	9.5	4.75	2.36	0.36	0.075
Specification limits	100	90-100	76-90	44-74	28-58	5-21	4–10
Passing (%)	100	94.4	76.6	49.8	33.4	21	8.2

Table 2: Grading of the aggregates used in this study.

Tests	Tests method	Results	Specifications	
	ASTM	-	Min	Max
Penetration of bituminous @ 25°C (100g ,5 Sec)	D5 [14]	48	40	50
Softening point of bitumen (ring and ball)	D36 [15]	54	50	60
Ductility @ 25°C	D113 [16]	135	>100	
Penetration of bituminous after heating	D5 [14]	35	-	
Ductility @ 25°C after heating	D113 [16]	107	>60	

Table 3: Results of asphalt binder tests.

Encapsulation procedure

The encapsulation procedure has been described in the following steps and briefly illustrated in the Fig. 2.

- In order to build the core of capsules the scoria porous sand was put in a tall container. Then, heavy vacuum slops (H.V.S), the rejuvenator was pourd until the height of rejuvenator-sand blend were twice as the original sand. Next, all of them were heated for 1 hour at 105 °C in order to decrease the viscosity of oil.
- The blend of the rejuvenator-sand in order to eliminate the air and force the oil was put into a vacuum chamber during at least 30 min. Heating and the vacuum process have been repeated twice to remove as much air as possible. After, the excess rejuvenator was eliminated; the blend was manually shaken in order to have a homogeneous blend.
- In order to build the shell, the blend of epoxy-sand-rejuvenators was manually mixed in a weight ratio 1:2.5 until the epoxy covered entire soil grains. In another round, epoxy in a weight ratio 1:20 (1 gram of epoxy for each 20 gram of rejuvenator-sand blend) was added. Next, cement was added fourfold as weight of the sand to the blend of epoxy-sand-rejuvenators. If the amount of cement were more than fourfold as weight of the sand, it would absorb all the epoxy and the oil inside the porous sand.

- The obtained capsules were added to a container of eight steel balls with a diameter of 2 cm in a volumetric ratio of 1:54 to the total volume of the container. Then, the container was energetically moved in circles for about 15 s.
- After forming the capsules' shape like ball, they were left to cure for 8 hour at 35 °C.
- In order to modify the wall of capsules, one part of nano-Zycosil was mixed with 100 parts of pure water. Capsules are immersed in the mixture for 2 to 5 s. Next, the capsules were stored at room temperature for 24 hours.

In order to evaluate the resistance of capsules during mixing process, they were incorporated into the asphalt mixtures as secondary aggregates. The mixture was blended during 15 min at 285 rpm at 160 °C in a 10 lit mixer and compacted using a Superpave gyratory compactor (SGC) to simulate a real porous asphalt pavement. Then, the sample was sawn in half and the cross section was examined.


a) b)
c) d)
e)

Figure 2: Encapsulation procedure step by step; a) Dry and cleaned porous sands (particle size about 2.3 to 2.8 mm), b) Porous sands with rejuvenator inside the grains, c) Container with a diameter of 2 cm steel balls, d) Energetically circles movement for 15 s, e) The sieved capsules between 3 and 4 mm.

Scanning electron microscopy (SEM) and computerized tomography (CT) scan analyses

Scanning electron microscopy (SEM) and computerized tomography (CT) scan analysis have been performed to analyse the capsule morphology. The SEM photos are taken at a voltage of 20.8 kV by a SEM microscope from Erbil asphalt laboratory in Iraqi Kurdistan.

Thermal analysis

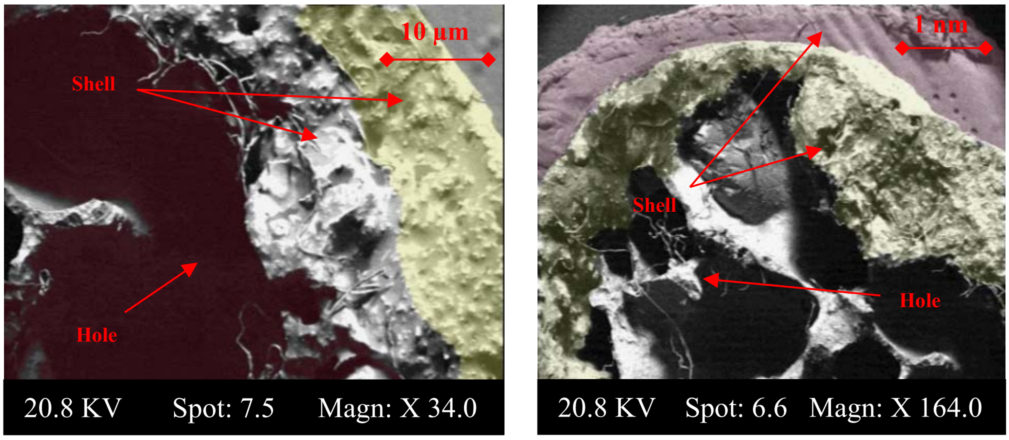
In order to evaluate the resistance of capsules during the mixing process of the asphalt mixtures, thermal gravimetric (TG) analysis was conducted. Thermo-microbalance of TG anlysis was performed using nitrogen atmosphere and heating rate of 10 °C/min. The compositions of capsules must resist temperatures between 160 °C and 180 °C. Moreover, these temperatures can produce molecular scissions or intermediate compounds in the rejuvenator such as ketenes or alcohol [17], as well as mass loss due to the evaporation of these compounds.

Indirect tensile strength (ITS) test

ITS test was conducted according to AASHTO-T283 [18] using the pneumatic load system in order to evaluate resistance of the asphalt mixtures samples against fracture and to determine their tensile strength. Marshal machine has been used to apply a compression load on asphalt concrete specimens at a rate of loading 51 mm/min, along a diametrical plane through two opposite loadings which curved strip heads of 12.5 mm width.

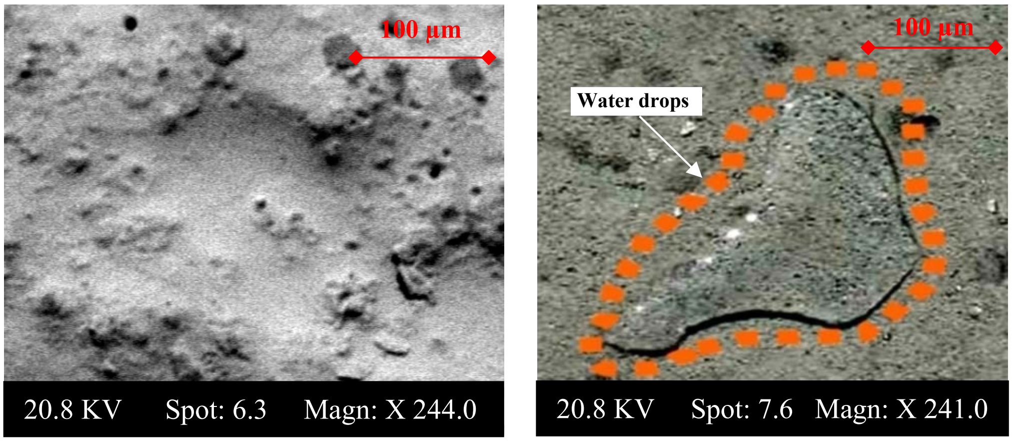
Indirect tensile fatigue (ITF) test

ITF test was done on three specimens for each studied asphalt mixture sample by applying a 0.2 s haversine repeated load followed by a rest period of 0.3 s at a frequency of 8 Hz. The maximum load applied at each cycle was 0.138 MPa. The equipment used was universal testing machine (UTM) according to ASTM-D4123 standard [19].



a) A section of the capsule

b) A section of the capsule



c) Water repellent of the shell

d) Water repellent of the outer wall

Figure 3: SEM images of capsule.

RESULTS

SEM and CT-Scan analyses results

SEM images of the capsules are displayed in Fig. 3. As it is illustrated in Fig. 3 a, micro pores in the capsule are clear. Also, shell part of the capsule with high density can be seen close to the porous sand. The shell part prevents the rejuvenator leakage from the capsule. Fig. 3 b, shows a section of the capsule including two distinct area namely: the core and the shell. Considering Fig. 3 b, it can be clearly seen that the shell has two different layers with variable density which are distinctly colored by Adobe Photoshop. In other words, the shell is completely made of a mixture of epoxy and cement, while the density significantly changes from inside toward outside of the capsule. In addition, during encapsulation procedure, the rejuvenator with low viscosity penetrated the wall of the capsule and changed the density of the shell from inside to the outside. Fig. 3 c, presents the water repellent of the shell due to modification of the capsule by nano-Zycosil. As it could be seen, water cannot penetrate the shell of capsule. Finally, as shown in Fig. 3 d, the outer wall of the capsule is well-repellent. In general, the shell well formed on the outer side. So, as an overall result, the shell of cement-epoxy prevents the leaving of rejuvenator to the outside of the capsule.

CT-Scan images of the six capsules are shown in Fig. 4. In this figure the bond between the porous stone and the shell looks very strong. It can be seen that externally they look like main aggregates because of their very uniform surface. Core and shell of the capsules are clearly seen in these figures. In general, the thickness of shell is directly related to the viscosity of rejuvenator. In other words, increase in viscosity resulted in increase in the thickness of wall and vice versa.

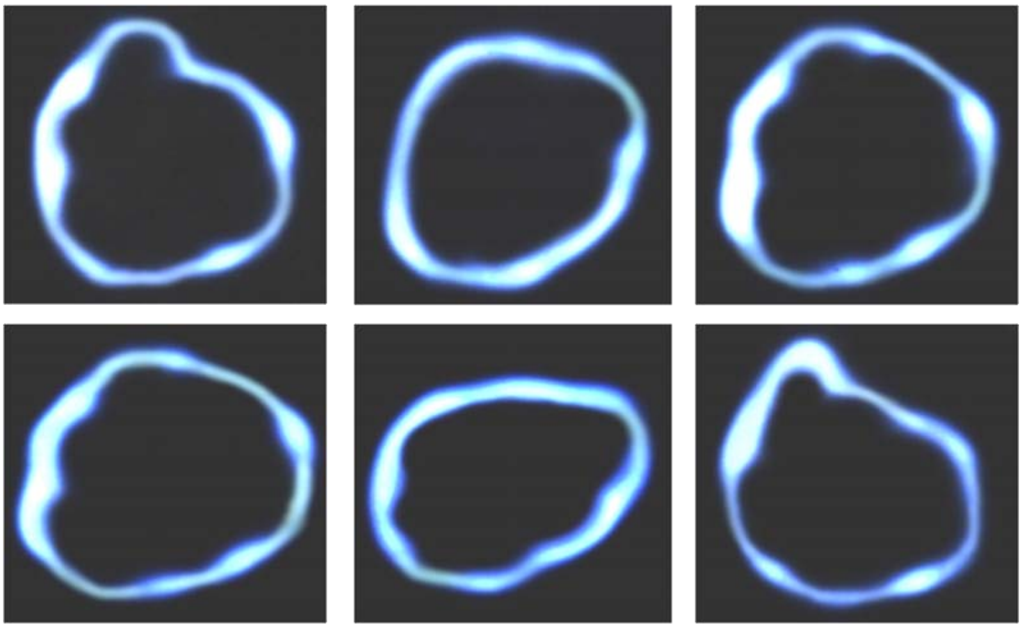


Figure 4: CT-Scan images of different capsules by shell thickness from 0.7 to 1.2 mm.

In order to ensure the integrity of the capsule's wall and preventing the rejuvenator leakage, some of them randomly were bisected before mixing to asphalt binder-aggregates. As shown in Fig. 5, the rejuvenator inside the capsule indicates the integrity of the wall.

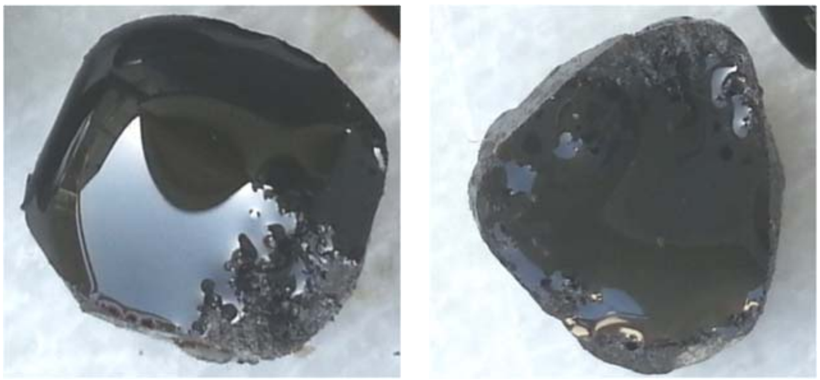


Figure 5: A bisected capsule between 3 and 4 mm diameter.

In order to evaluate the resistance of capsules during mixing process, they were incorporated into the asphalt mixtures as secondary aggregates. Then, as it is displayed in Fig. 6 a, the samples were sawn in half and examined with by naked eyes. According to Fig. 6 a, the capsules had high resistance during mixing process and still had lots of oil in them. In this figure the capsules are integrated in the matrix of Asphalt binder-aggregates-capsules as secondary aggregates. On the other hand, considering Fig. 6 b, the capsule broke under the stress caused by condensation.

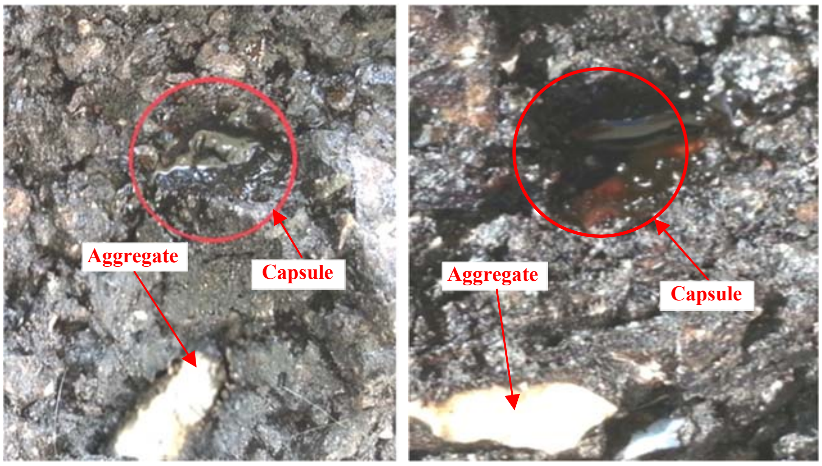


Figure 6: Capsules with thickness from 3 to 4 mm embedded in asphalt mixture. a) Capsule resisted the mixing process; b) Capsule broke under the stress by condensation.

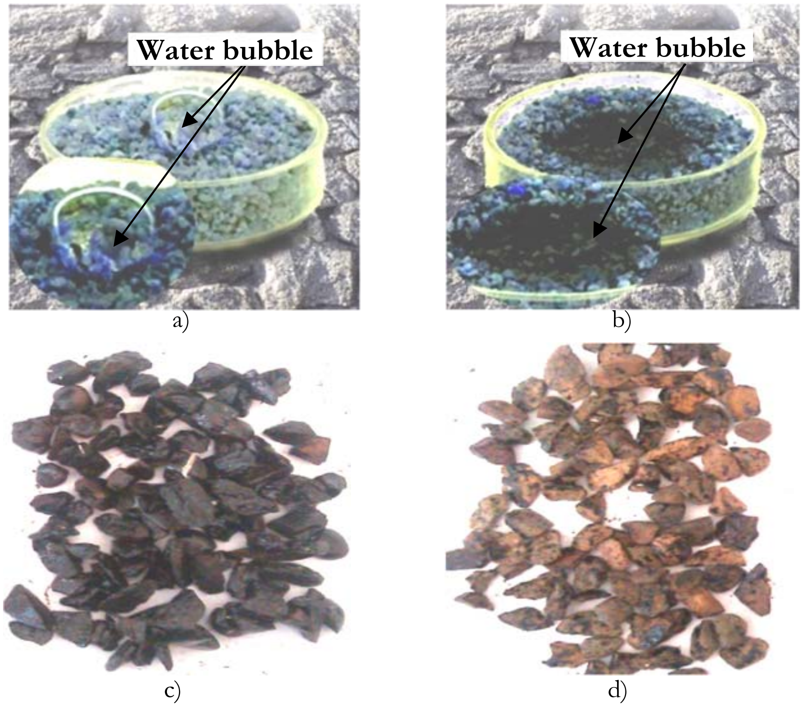


Figure 7: a) Hydrophobicity of the aggregates modified with nano-Zycosil; b) Hydrophilic in the unmodified aggregates; c) Amount of stripping in the modified aggregates (with grading according to Tab. 2); d) Amount of stripping in the unmodified aggregates (with grading according to Tab. 2).

In Fig. 7 a, the aggregate is shown after modification with nano-Zycosil. As shown in Fig. 7 a, nano-Zycosil modification led to creating a hydrophobic surface on the aggregates as the drop retained its shape and did not penetrate the aggregates. On the other hand, considering Fig. 7 b in the normal state the surface of aggregates is hydrophilic and although holes in the aggregates are smaller than water molecules, but when the water molecules collide with the surface, atoms would break and water penetrate to the holes. Moreover, one of the reasons is the presence of silica in the surface of the aggregate which make their surfaces hydrophilic. By modifying the surface of aggregates with nano-Zycosil, this material due to their nano-meter size, penetrated into the pores of the aggregate and provided the hydrophobic properties. When the water collided the surface of the modified aggregates, it remained on the surface of them. Figs. 7 c and d, are taken after water boiling test and indicate the amount of stripping for modified and unmodified aggregates with 1% nano-Zycosil, respectively. The amount of stripping in the unmodified ones is significantly much more than the modified aggregates [20].

Thermal analysis results

Fig. 8 shows TGA diagrams for the capsule components. As it is illustrated in Fig. 8, the mass loss of rejuvenator, nano-Zycosil and epoxy resin starts to fall down at 230°C, 190°C and 260 °C, respectively. As mentioned before, the asphalt mixture mixing temperatures was between 160 and 180 °C. This means the capsule can resist the asphalt mixture mixing temperature range. In addition, Fig. 8 also shows, the most affected material by temperature is the scoria porous sand which lost a lot of mass between 30 and 160°C. That is because of the presence of water in its pores. So, to avoid such mass loss it is required before encapsulation, the porous sand should be heated in order to evaporate the moisture in its pores.

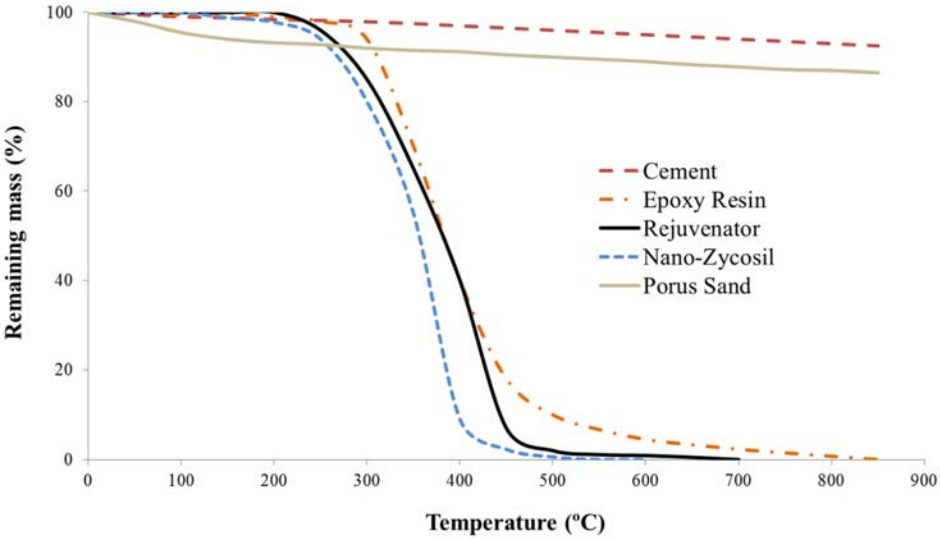


Figure 8: TGA diagrams of encapsulated ingredients.

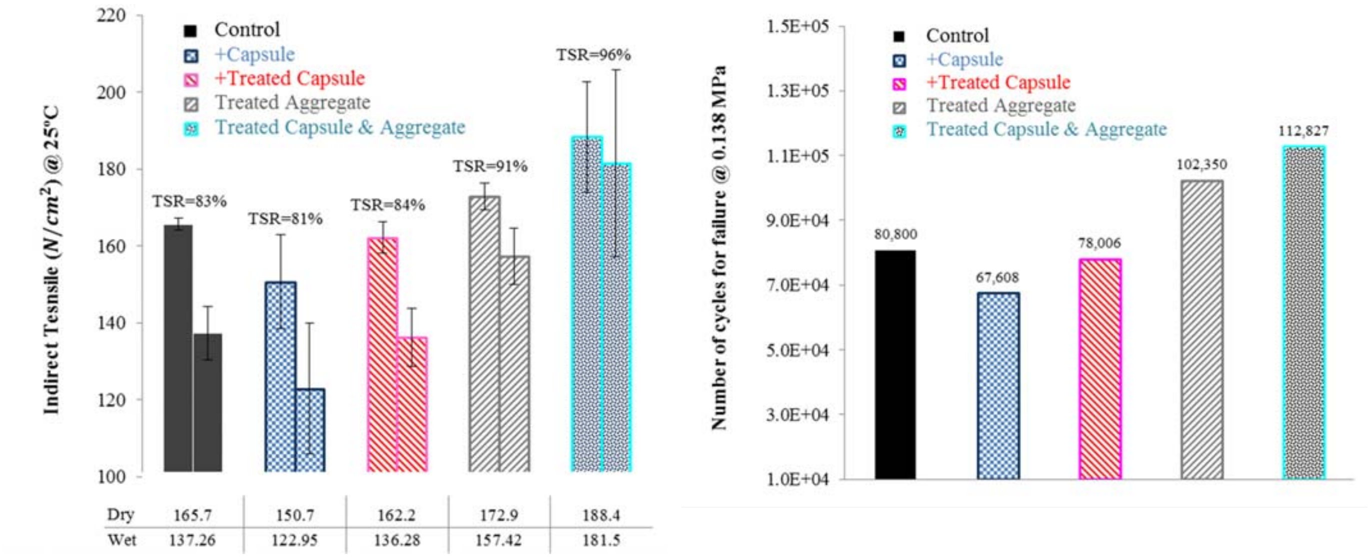
ITS and ITF tests results

In Figs. 9 a and b, results of ITS and ITF tests are shown, respectively. In Figs. 9 the prefix “Treated” means the material is modified with nano-Zycosil. As it is displayed in Fig. 9, different types of samples including asphalt mixture without capsules, with capsules, with modified capsules, modified aggregate and with modification of both capsules and aggregates by nano-Zycosil were experimented. According to Fig. 9 a, it can be seen that the capsules reduce the stiffness of asphalt mixture and have some effect on the mechanical characteristics of the asphalt mixture. This could happen because some of the capsules broke during the mixing. Another reason could be the diffrences in the adhesion between them and the asphalt binder compare to the adhesion between the main aggregates and the asphalt binder. In addition, during the mixing process, some of the capsules would break which results in releasing the rejuvenators. So, the released rejuvenators would soften the asphalt binder.

Moisture susceptibility of asphalt mixtures could be predicted by tensile strength ratio (TSR), wet/dry (conditioning/ unconditioned) ratio of ITS values [18]. In general, the higher the TSR, the more resistance mixture against moisture induced distresses such as stripping. As shown in Fig. 9 a, the mixtures modified with nano-Zycosil had lower susceptibility against wet condition due to having higher TSR values. Treated Capsule & Aggregate-mixtures had the highest moisture resistance while +Capsule-mixtures performed the worst among the others. As a result, modified capsules and aggregates with nano-Zycosil improved the moisture resistance of asphalt mixture significantly regarding TSR values.

Also, the results showed that by modification of the capsule and aggregates, the indirect tensile and fatigue strength of specimens increased. That is because, without modification with nano-Zycosil, the adhesion between the asphalt binder and the surface of the capsules very weak. By modifying the capsules and aggregates with nano-Zycosil, the adhesion between the capsules and aggregates with asphalt binder converted from silanolian to siloxane types. In general, the seloxane bonding is much stronger than silanolian one. So, due to this conversion the percent of asphalt binder components which would contribute to the matrix of capsules-aggregates-asphalt binder increased from 40-45 % to 85-95 %.

After modification of the aggregates, aggregates and capsule with nano-Zycosil, the samples were examined in a two-phase evaluation. The results showed that the modification of aggregates with nano- Zycosil in the samples including unmodified capsules significantly increased tensile strength and fatigue resistance compare to samples without modified aggregates. It indicated that modification of aggregates with nano-Zycosil had great impact on indirect tensile strength and fatigue resistance of asphalt mixture samples.



a) b)

Figure 9: The results of ITS and ITF tests. a) The indirect tensile strength of specimens; b) Number of cycles required to to break specimens.

Another part of the results of ITS and ITF tests are illustrated in Fig. 10. Same as for Figs. 9, in Figs. 10 the prefix "Treated" means the material is modified with nano-Zycosil. In Fig. 10 a, the displacement of specimens under the loading at un-conditioning state before and after the fracture is illustrated. As it was found, displacement of samples with capsules are more than the samples without capsules. That is because of the adhesion resistance between the capsules and asphalt binder is less than the adhesion resistance between the main aggregates and the asphalt binder. This caused a significant increase in the displacement of the samples including the capsules compare to the ones without the capsules. In addition, the samples modified with nano-Zycosil had less displacement than the ones which did not modify with nano-Zycosil.

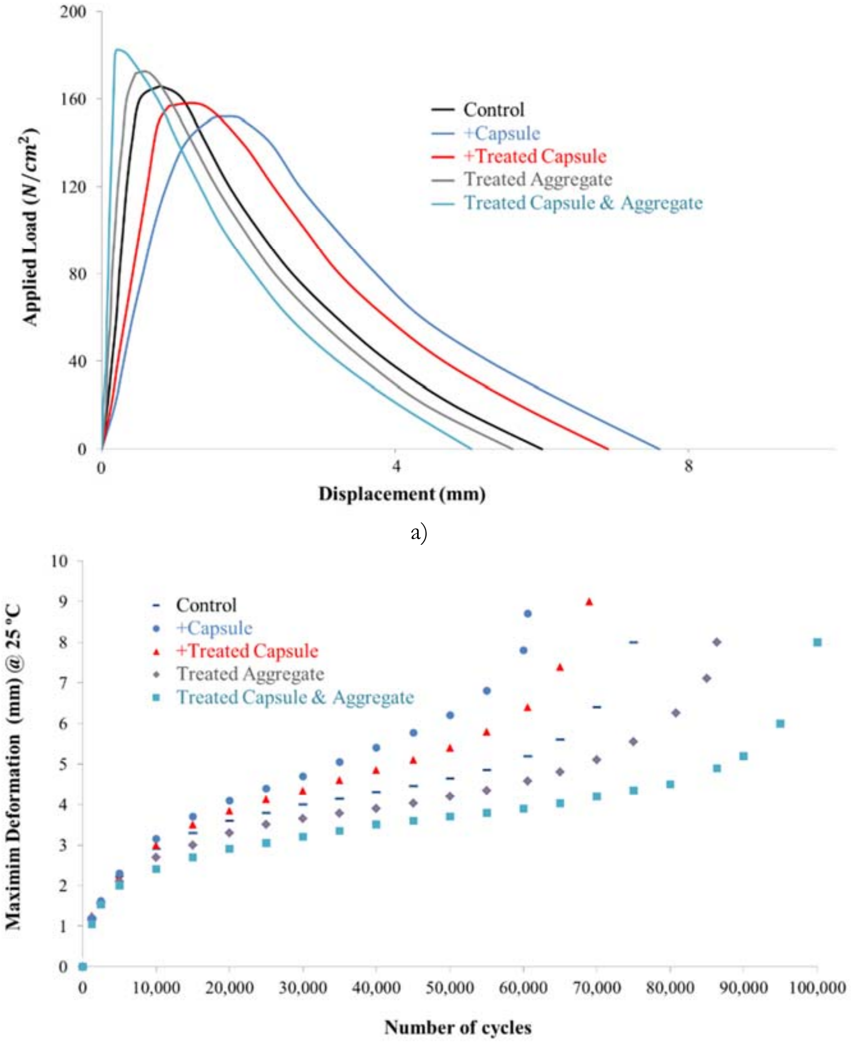
Fig. 10 b, presents the values of deformations under the fatigue loading until the fracture was indicated. As it is displayed in Fig. 10 b, the effect of the modified capsules, modified aggregate and both together are so different. Modification of the capsules by nano-Zycosil reduced the deformation compare to the unmodified capsule samples. Moreover, modified aggregate and modified capsules together samples had the least amount of deformation and the most amount of number of cycle of failure.

CONCLUSIONS

The following conclusions can be deduced from the obtained results:

- Considering the morphology evaluation and the TGA diagrams, it was found that most of the capsules resisted the mixing procedure of the asphalt mixture. So, the encapsulation procedure used in this study was a successful technique to build rejuvenator capsules. On the other hand, some of them broke under indirect tensile tests and reduced the stiffness of the asphalt mixtures.

- By adding the capsules to the asphalt mixture samples, fatigue resistance and deformation decreased and increased, respectively, because of less interlock between the capsules and asphalt binder compare to aggregates. On the other hand, modification with nano-Zycosil as an anti-stripping agent significantly improved the adhesion strength in the matrix of capsules-aggregates- asphalt binder by converting the adhesion type from silanolian to siloxane.



a)

b)

Figure 10: The results of ITS and ITF tests. a) Displacement of specimens under indirect tensile load; b) Maximum deformation of specimen during the loading Until the samples broke.

- Obtained results demonstrated the increase of TSR by implementation of modified capsules and aggregates with nano-Zycosil. For instance, TSR significantly increased about 14% for Treated Capsule & Aggregate-mixtures compared with the conventional asphalt mixtures. On the other hand, the mixtures with modified capsules by nano-Zycosil had 5% more TSR than the ones with unmodified capsules.
- It was found that the capsules alongside main aggregates both modified with nano-Zycosil improved resistance of studied mixture against moisture damage by improving the adhesion in the matrix of capsules-aggregates-asphalt binder.
- Overall, modification of capsules and main aggregates together with nano-Zycosil significantly improved mechanical performance of the asphalt mixture samples.

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