Effect of nanomaterials on ageing and moisture damage using the indirect tensile strength test

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Abstract

Environmental conditions as well as traffic loads lead to the deterioration of asphalt pavements during their service life. For this reason, the use of nanomaterials that improve the mixtures behaviour could be interesting. The behaviour of two mixtures made with binder modified with nanoclay and nanoiron, and their strength against ageing and moisture damage is studied. Mixtures have been subjected to ageing by two procedures: extended heating, Long-Term Oven Ageing (LTOA), and ultraviolet (UV) plus rainfall simulation, Tecnico Accelerated Ageing (TEAGE). The results show that nanoclay improves the mixture behaviour against ageing, while nanoiron does against moisture damage.

Keywords: ageing, weather ageing simulation, nanoclay, nanoiron, moisture damage

1. Introduction

The main factors affecting the durability of asphalt mixtures are ageing and moisture damage, assuming the pavement is well constructed [1, 2]. This is why it is crucial to take into account the effect of the environmental conditions on the asphalt mixtures when designing them. Simulating the effect of these factors on asphalt mixtures is a complicated task as many of them such as temperature, water, air, solar radiation, and their combination influence on the durability of mixtures.

Ageing, phenomenon by which the behaviour of the binder and of the mixture changes, is an important factor in the context of cracking resistance of asphalt mixtures [3]. This phenomenon is characterized by a binder hardening, which increases the stiffness values of the mixture. This means, on the one hand, an increase of the mixture bearing capacity and, on the other hand, an increase in its resistance to deformation. However, these effects occurred just to a certain point once ageing also means fragility of the binder/aggregate bonding inducing much less resistance to fatigue and enhancing the aggregates' stripping.

Ageing is generally divided into two stages [4]: short-term ageing and long-term ageing. The short-term ageing occurs during the manufacturing, mixing, transport and laying of the asphalt mixture. During this stage, ageing is mainly associated with the loss of volatile components and the oxidation of bitumen. On the other hand, long-term ageing is associated exclusively with the degradation produced during the service life of the mixtures as a result of environmental factors such as temperature, available oxygen in the atmosphere, UV radiation and moisture [5]. In this stage, the oxidation rate decreases with the depth of the asphalt layer [6, 7]. Consequently, the viscosity also decreases with the asphalt layer depth [8]. This is due to a constant supply of oxygen, the high temperatures of the surface layer and UV photo-oxidation.

Evaluating and characterizing the effect of ageing on the behaviour of asphalt mixtures is a difficult task [9]. The basic procedure is to age artificially the mixture and then evaluate the effect of ageing through simplified approximations that relate to a mechanical property of the material. The most commonly methods used to evaluate ageing are dynamic modulus, diametrical resilient modulus and lost of ductility. Other methods related to the study of asphalt mixtures cracking such as the indirect tensile strength test, the semi-circular bending (SCB) fracture test or the Fénix test have been used to determine the effect of ageing [10, 11, 12]. However, there is not a good number of studies examining the effect of ageing on the indirect tensile strength, being an unresolved problem at present [10].

Throughout the literature, a number of methods have been defined for artificially ageing asphalt mixtures. However, the correlation of these methods with field data is extremely difficult as it depends on many factors such as the location of the road, the sun exposure, the weather, the type of mixture and the void content. Airey [1] divided the ageing methods for asphalt mixtures into four categories: prolonged heating procedures, oxidative procedures, ultraviolet and infrared light treatments and steric hardening. The simulation of ageing in the laboratory is usually carried out, especially for binders, under the influence of temperature and pressure. This differs from the conditions at which mixtures are aged in the field. One of the most followed ageing methodologies by the scientific community is the defined by the SHRP program [13], method on which AASHTO R 30 is based. The laboratory ageing procedure for asphalt mixtures defined by the SHRP program consists of the Short-Term Oven Aging (STOA), where the loose mixture is placed in a convection oven at 135ºC for 4 hours. Then, the mixture is compacted and placed in a convection oven at 85ºC for 5 days (Long-Term Oven Aging, LTOA). Nevertheless, a method that takes into account environmental factors such as temperature, UV radiation and moisture (simulation of rain conditions) should be considered to perform a more realistic study of ageing [14, 15]. In order to simulate the effect of daylight as well as the radiation from the sun, ultraviolet radiation treatments have been used in the literature. In this respect, some authors have used the wetherometer test that combines the effect of the temperature, the UV radiation and the moisture during ageing to simulate the service conditions of the mixtures [14, 16, 17, 18]. The results show the importance of ultraviolet radiation in the ageing of the asphalt mixtures [18, 19].

The reduction in the asphalt mixture resistance is not only a result of ageing but also of moisture damage, contributing to the development of several types of pavement deterioration such as cracking, rutting and collapse [20, 21, 22]. As a consequence, the pavement life decreases [23].

Moisture damage is normally associated with five different mechanisms: detachment, displacement, spontaneous emulsification, pore pressure and hydraulic scour [24, 25, 26, 27, 28, 29]. Authors such as Hamzah, et al. [30] add the effect of environmental conditions as a contributing mechanism to moisture damage. In addition to the above mechanisms, Kinggundu & Roberts [31], Little & Jones [32] and García [33] include the pH of water.

The development of tests to determine the moisture sensitivity of asphalt mixtures began in the 1930s [34]. Since then numerous procedures have been developed in an attempt to identify the asphalt mixture susceptibility to moisture damage. Generally for laboratory tests on compacted mixtures, specimens are conditioned in water to simulate service conditions. The moisture damage assessment is measured by the ratio between the stiffness or strength of the conditioned and the unconditioned mixtures. Some of these tests are the immersion-compression test [35, 36] and the Marshall stability test [35].

Currently, the study of the asphalt mixtures properties during their service life has led to the implementation of different test methods, which intend to improve the quantification of ageing together with the moisture damage. Thereby, researchers from the Nottingham Transportation Engineering Center (NTEC) developed the Saturation Ageing Tensile Stiffness (SATS) test that combines oxidative ageing along with the moisture damage in the specimens conditioning [37, 38, 39].

Over time, asphalt pavement layers are exposed to great stresses, in addition to the effect of the continuous climate changes, which leads to the deformation of the lower asphalt layers, the stiffness of the upper asphalt layers and the relative displacement of the mixture mineral particles of the upper layers [40]. For this reason, the use of materials (nanomaterials) that modifies and ultimately improves the behaviour of mixtures becomes interesting [41]. This is why the use of nanomaterials is currently being studied. Different types of nanomaterials have been used to improve the behaviour of mixtures such as carbon nanotubes, nanoclay or nano-SiO2 [42]. Thus, the nanoclay can be applied to improve the mechanical behaviour and the resistance to ageing of the asphalt mixtures [43, 44].

In this study, the effect of ageing by UV radiation and moisture damage on the asphalt mixtures cracking has been investigated. For this purpose, unconditioned and aged mixtures, subjected or not to moisture damage, are tested by the indirect tensile strength to analyse their cracking resistance and their sensitivity to moisture damage. Ageing by

UV radiation has been carried out with a new test protocol developed by the Instituto Superior Técnico (IST), TEAGE (from the acronym of TEcnico Accelerated aGEing). This test takes into account not only the UV radiation but also the effect of the rain on the asphalt mixtures, adapted for the total conditions observed for a specific site, meaning that UV and rain simulation incidence over mixtures are the same that the ones verified in average during a certain number of years in that site. In addition, the results from the indirect tensile strength performed on the mixtures aged with the TEAGE procedure are compared with those obtained on mixtures aged with the procedure defined by the SHRP, LTOA. Furthermore, the effect of modified bitumen with nanoclay and nanoiron on ageing and moisture damage is analysed.

2. Methodology

2.1. Materials and sample preparation

An asphalt concrete mixture with a maximum aggregate size of 14 mm (AC14) was selected. The aggregate type was granite with limestone filler. The AC14 aggregate gradation is presented in Figure 1. Specimens were prepared with different binder: a conventional one (35/50), a nanoclay modified one and a nanoiron modified one. For all the cases, the binder content was 4.5% by the weight of the mixture. The modification of both nanomodified bitumen was performed by mechanical dispersion of the nanoparticles in the neat binder using a high speed stirrer. For the two nanomodified bitumen, the nanoparticles content was 4% by weight of modified binder.

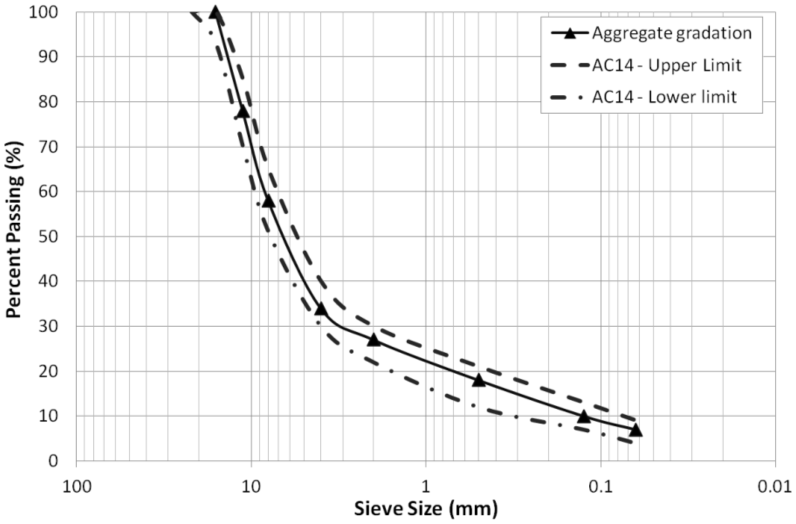

Figure 1. Aggregate gradation of AC14 mixture.

The nanoclay is a hydrophilic bentonite ($H_2Al_2O_6Si$). The particles of this material form agglomerates with an average size of less than 25 µm, has a density of 2400kg/m^3 and pH in the range of 6 to 9. The nanometric dimension of the nanoparticle is determined by its thickness and the spacing of the laminar structures it forms (between 1 and 2 nm).

The iron nanoparticles have an average size of less than 50 nm. These are nanoparticles with zero valence electrons, Fe (65 to 80%), and iron oxides, FeO and Fe_3O_4 (20 to 35%). This material has a specific surface area of 25 m^2/g, density from 1150 to 1250 kg/m^3, and alkaline properties (pH from 11 to 12).

Cylindrical samples of asphalt mixture (101.6 mm diameter and 64 mm height) were prepared using the impact compactor (Marshall hammer). The specimens were compacted according to EN 12697-30:2012 [45] applying 50 blows on each side of the cylindrical sample. The mixing temperature was 165ºC and the compaction temperature was 155ºC. The bulk density of the mixtures as well as the theoretical maximum density were determined following the EN 12697-6:2012 [46] and EN

12697-5:2009 [47], respectively. Table 1 shows the average percentage of air voids of the mixtures before its conditioning (ageing and/or moisture damage). As it can be seen the mixtures with nanoparticles have a lower level of air voids. This fact is probably due to the fact that viscosity of the modified binder increases because of the addition of nanoparticles [41]. An increase in the viscosity could lead to an increase in the binder film thickness that coats the aggregates, which could entail the decrease in the level of air voids.

Table 1. Average percentage of air voids.

Mixture	Air Voids (%)	Standard Deviation
Control	7.6	0.9
Nanoclay modified	6.2	1.1
Nanoiron modified	5.3	0.8

Figure 2 shows an overview of the experimental plan carried out in this study. The experimental plan has been divided into three groups of specimens: unmodified mixture specimens, nanoclay modified mixture specimens and nanoiron modified mixture specimens. Each one is divided into three: the fresh asphalt mixture specimens, the specimens aged by TEAGE procedure and specimens aged by the SHRP Long-Term Oven Aging protocol. Finally, half of the specimens from each group have been subjected to moisture damage according to EN 12697-12:2008 [48].

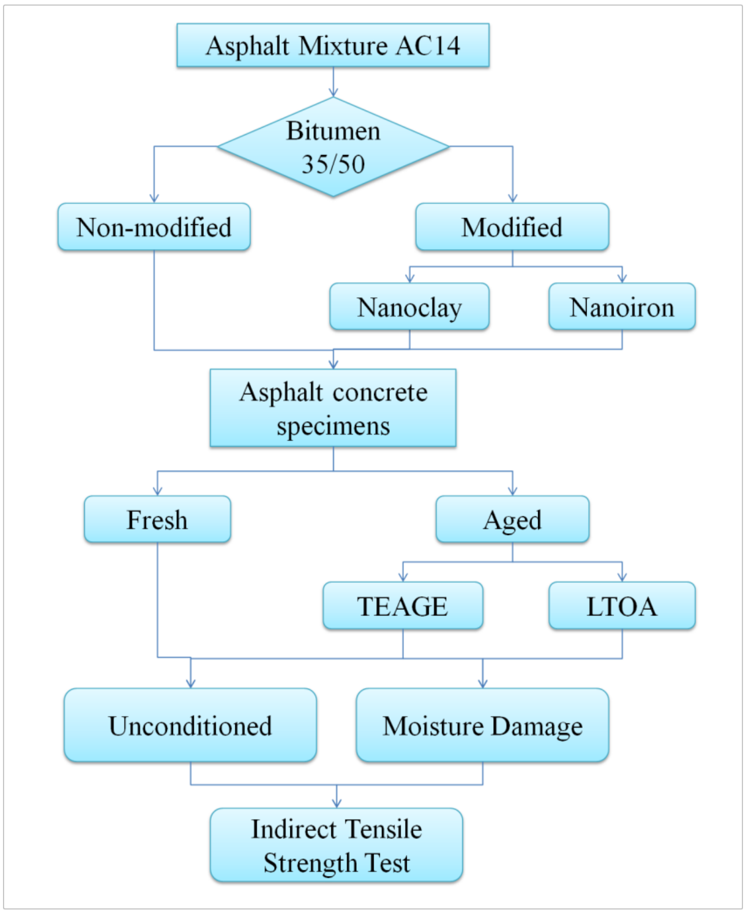

Figure 2. Experimental plan.

2.2. Ageing conditioning

Two different methods to age mixtures, TEAGE and LTOA, have been applied in order to analyse the effect of ageing and validate the results of the TEAGE method.

The TEAGE is a procedure to age asphalt mixtures due to their exposure to UV radiation and to moisture [15]. The main objective of this procedure is to simulate the environmental conditions at which pavements are exposed during their service life. In this study, as stated, was used to simulate 7 years of weather exposure in Lisbon region (considering for 7 years the energy received by UV radiation and the total number of days with precipitation over 5 mm). The specimens where conditioned inside a special chamber where, using weather historic data, UV radiation and watering/drying cycles were applied (Figure 3).

Figure 3. Specimens being subjected to TEAGE ageing.

On the other hand, the Long-Term Oven Ageing procedure, following the SHRP programme A-383 [49], consisted of keeping the asphalt mixture specimens in the oven at 85ºC for 120 h (5 days). This long-term ageing process means 5 to 10 years of average climate incidence for the USA average conditions [50].

2.3. Moisture conditioning

Specimens have been conditioned according to EN 12697-12:2008 [48] related to the water sensitivity analysis of asphalt mixtures. The procedure consists of placing the set of specimens in a water bath at 40ºC for a period of 72 h. The wet set of specimens is previously subjected to vacuum with an absolute pressure of 6.7 kPa for a period of 30 min.

2.4. The indirect tensile strength test

The indirect tensile strength (ITS) is defined as the maximum tensile stress calculated as a function of the peak load and the dimensions of the specimen. The ITS of the specimens has been calculated from the indirect tensile strength test defined in the EN 12697-23:2003 [51], Equation 1.

$$ITS = \frac{2P}{\pi DH} \tag{1}$$

where ITS is the indirect tensile strength (GPa), P is the peak load (kN), D and H are the diameter and the height of the specimen (mm), respectively.

At least four specimens were tested for each condition and per mixture type. The test temperature was 15ºC as done in other studies [2].

The indirect tensile strength ratio ($ITSR$) is calculated as the ratio, in percentage, of the ITS of the wet set of specimens (ITS_{wet}) versus the ITS of the dry set of specimens (ITS_{dry}), Equation 2.

$$ITSR = \frac{ITS_{wet}}{ITS_{dry}} \times 100 \tag{2}$$

A similar parameter is defined to analyse the effect of ageing on the indirect tensile strength. This parameter is called Ageing Index [52] and is calculated as the relationship between some property measured in the aged mixture and the same property measured in the unaged mixture. In this case, the measured property is the indirect tensile strength (equation 3).

$$Ageing\ Index = \frac{ITS_{aged}}{ITS_{unaged}} \tag{3}$$

3. Results and discussion

Figure 4 shows the average values of the indirect tensile strength (ITS) for the different case studies. For the unconditioned mixtures (dry-fresh), the values of the indirect tensile strength increase when the mixture is modified with nanomaterials. This increase is higher for the nanoclay modified mixture. Thus, the physical dispersion and chemical reactions between the nanoclay and the binder have a significant effect on the internal structure of the mixtures, and on their rheological behaviour, which in general lead to an improvement on mechanical characteristics (e.g., fatigue and dynamic modulus) and on other properties (affinity binder-aggregate).

The surface area of binder also improves when nanoiron particles are dispersed in it. This could induce to an increase of the adhesion energy between aggregates and binder, increasing their adhesive strength due to cation exchanges in the modified binder.

When fresh mixtures are subjected to moisture under the conditions defined in EN 12697-12:2008 [48], the values of ITS decrease for all cases. For the modified bitumen mixtures, the ITS values after immersion are higher than the ones for the reference mixture. Values are very similar for the two mixtures modified with nanomaterials.

When the mixtures are aged, the ITS values increase due to the binder hardening. In the case of aged mixtures by the LTOA procedure, the ITS values of the modified mixtures are greater than the one of the reference. As for the fresh and dry condition, the higher values are found for the nanoclay modified mixture. When mixtures aged by the LTOA procedure are subjected to moisture damage, the highest ITS values are found for the nanoiron modified mixture. As well as dry aged mixtures by the LTOA procedures, the ITS values are greater for the modified mixtures.

As the mixtures are aged by the TEAGE procedure, the modified mixtures again have a higher strength than the reference one. The ITS value is approximately similar for both mixtures. This indicates that modified mixtures have a similar behaviour against UV ageing. When these mixtures are subjected to the action of water [48], the modified mixtures ITS values are higher than the unmodified one. The nanoiron mixture presents a higher value than the nanoclay modified one as the case of dry mixtures.

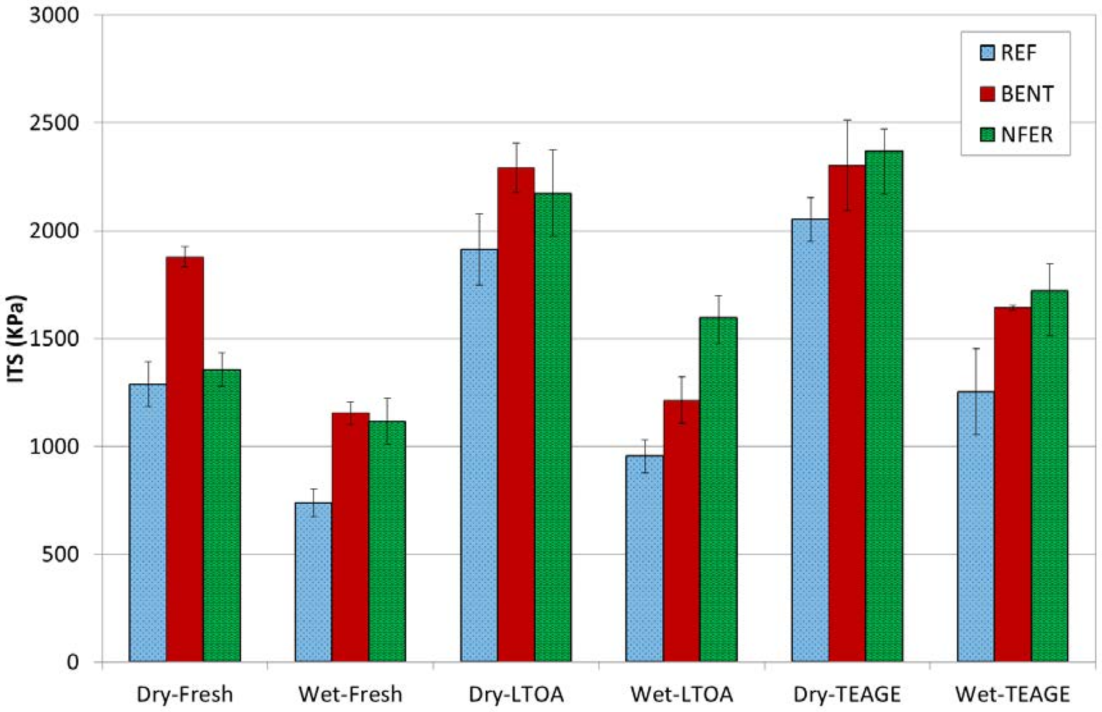

Figure 4. Average values of the Indirect Tensile Strength (ITS) for the case studies.

Effect of the action of water

The indirect tensile strength for the three mixtures analysed and for the ageing conditions decreases significantly after subjecting the mixtures to moisture damage [48]. However, differences in the behaviour of the mixtures against the action of water are observed. To study these differences in more detail, the water sensitivity of the mixtures expressed by the Indirect Tensile Strength Ratio (ITSR) is calculated.

Figure 5 shows the values of ITSR for the different case studies. At first, it is noticed that the reference mixture has a low ITSR value. This could be due to the type of aggregate (granite) and the air void content of the mixture (greater than 7%). When fresh mixtures are subjected to moisture damage, the nanoclay modified mixture exhibits a similar behaviour to the reference mixture. On the other hand, the nanoiron modified mixture presents a better behaviour against moisture damage: the decrease in the indirect tensile strength is lower, which corresponds to an important increase in the value of ITSR. This value increases by more than 20% in relation to the reference mixture.

In the case of mixtures aged with LTOA, the indirect tensile strength values follow a similar trend to the fresh mixtures. ITSR values of the reference mixture and the nanoclay modified mixture are similar, increasing for the nanoiron modified mixtures. For the three mixtures, ITRS values are lower than the obtained for fresh mixtures. Therefore, ageing conditioning causes a decrease in the resistance of the mixtures against moisture damage.

When the mixtures are aged with the TEAGE procedure, the ITSR values of the modified mixtures are higher than those of the reference one, both presenting similar ITSR values. After TEAGE ageing, the reference mixture for the fresh condition seems to present the same behaviour while the nanoclay modified mixture has improved and nanoiron mixture has decreased.

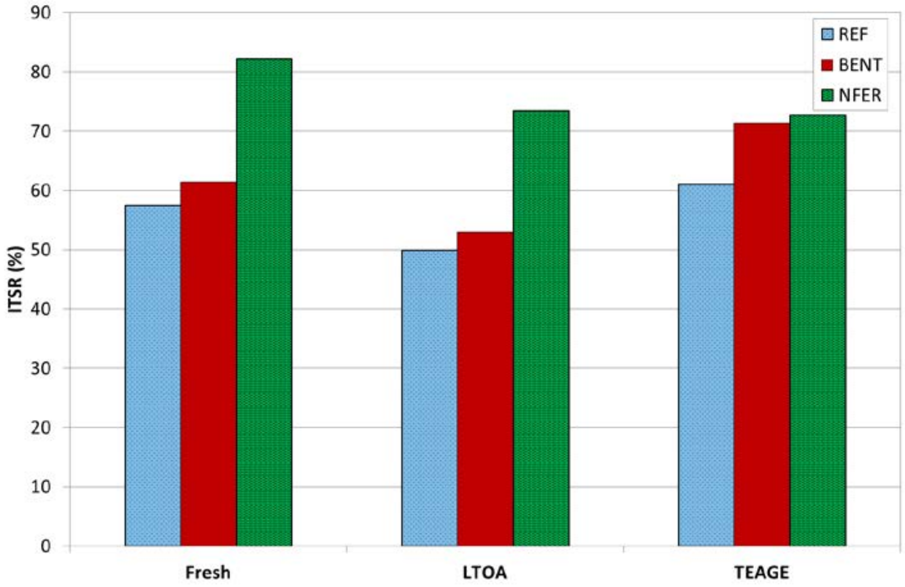

Figure 5. Values of the Indirect Tensile Strength Ratio (ITSR) for the case studies.

Effect of ageing

The influence of ageing on the indirect tensile strength is analysed by the so-called Ageing Index, computed according to equation (3), and is showed on Figure 6. Values of the index close to 1 indicate that the effect of the type of ageing used on the indirect tensile strength of the mixture is less evident. All the values are above 1, showing that the effect of the type of ageing (hardening the binder) has effect on samples. In all cases, the effect on hardening the binder is less effective for the nanoclay modified mixture. This indicates that nanoclay modification probably induce flexibility for the mixture during more years. However, this effect is less expressive when mixtures are subjected to water conditioning [48] and to the TEAGE process. The opposite was verified for the LTOA process.

When mixtures are aged by TEAGE, these rates are higher. This fact may be indicating that mixtures are more susceptible to TEAGE (6 years of Lisbon climate) ageing than

LTOA (ten years for USA average climate). As in the previous case, the closest values to 1 are found for the nanoclay modified mixture.

After subjecting the aged mixtures to moisture damage, the values of the ageing index tend to decrease, except for the reference mixture and the nanoclay modified mixture aged by TEAGE procedure where values increase. In the case of ageing by LTOA, the value of the ageing index for the nanoclay modified mixture after immersion is close to 1.

After subjecting the mixtures aged by TEAGE to the action of water [48], the ageing index values of the reference mixture and the nanoclay modified mixture increase compared to those obtained for the dry condition. On the other hand, the values for the nanoiron modified mixture decrease. These results indicate that moisture has a significant influence on the behaviour of aged mixtures, obtaining different results depending on whether or not mixtures are subjected to moisture damage [48].

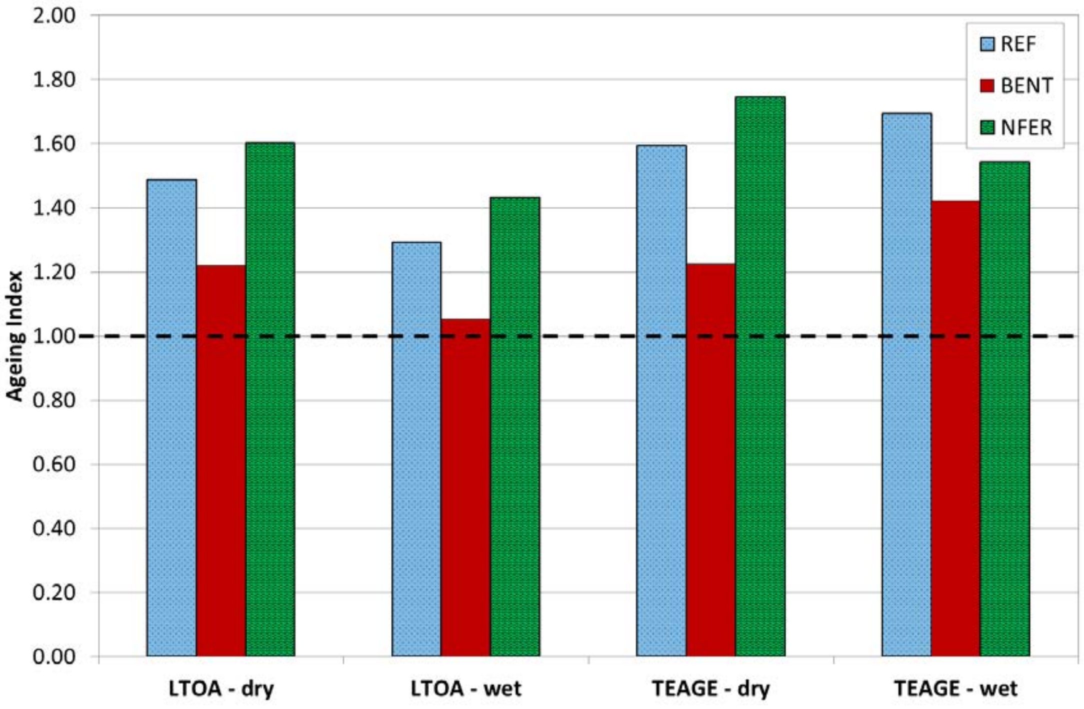

Figure 6. Values of Ageing Index for the case studies.

Combined effect of ageing and the action of water

In order to analyse the combined effect of ageing and the action of water in the mixtures, the ratio between the indirect tensile strength (ITS) of the wet aged mixtures and the ITS of the same mixture, dry fresh (not wet and not aged), have been obtained and denoted as $ITSR_{wa/df}$, where the subscript wa/df stands for wet aged over dry fresh. Figure 7 shows the results of this parameter for the two ageing procedures. In general, the combined effect of these two phenomena causes a decrease in the strength values, except for the case of nanoiron modified mixtures.

In the case of mixtures aged by LTOA, the $ITSR_{wa/df}$ values are lower for the nanoclay modified mixtures, and greater than 100% for the nanoiron modified mixtures. For this kind of mixture, the increase in strength due to ageing without loosing bind capacities, and because of this, is higher than the decrease caused by the action of water. This indicates that this type of mixture probably deals better with ageing, increasing strength to a certain extent without loosing consistence and bond aggregate/binder properties, being is this way more resistant to moisture aggression during the life cycle.

When mixtures are aged by TEAGE procedure, the nanoclay modified mixture slightly decreases its $ITSR_{wa/df}$ value compared with the reference, while the nanoiron modified mixture exhibits an $ITSR_{wa/df}$ value greater than 100%. Two inferences could be made: the nanoiron modified mixture behaved as was described for the LTOA process; the nanoclay modified mixture confirm with TEAGE the same as with LTOA, that it is slightly more affected by the ageing process than the reference mixture, which indicates a less applicable mixture modification with respect to the combined effect of ageing and the action of water.

Comparing both ageing procedures, a similar trend is observed. For all mixtures, the $ITSR_{wa/df}$ values increase when the TEAGE ageing procedure is used, indicating that the 6 years of climate simulation for Lisbon over the mixtures are less destructible than the LTOA process, actually not applicable to the Lisbon climate. This indicates the susceptibility of the mixtures to the use of different ageing procedures and the necessity of an ageing process attached to the region where the mixtures will be used.

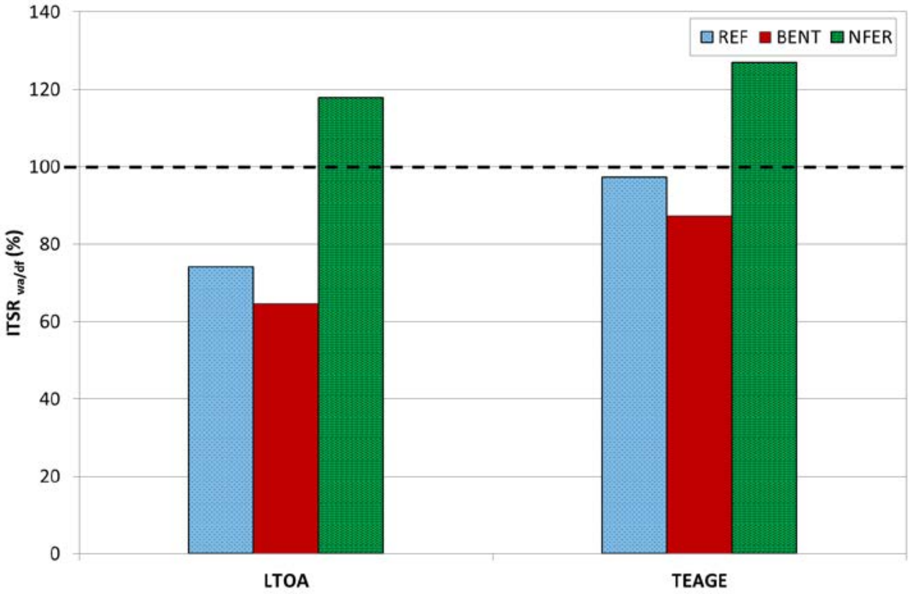

Figure 7. Values of the Indirect Tensile Strength Ratio ($ITSR_{wa/df}$) between wet aged mixtures and dry fresh mixtures.

The load-displacement curve of the indirect tensile test allows the displacement values when the maximum load is reached in the test to be obtained. With this value, it is possible to analyse the deformation the mixture is able to withstand before breaking. Figure 8 shows the average values of the displacement for the different cases studied.

When the mixtures are unconditioned (fresh), differences are observed in the displacement values depending on the type of binder. The values are higher when the mixtures are modified; obtaining the highest values for the nanoiron modified mixture. After moisture conditioning, the displacement values fall. This is expected once the ITS also fall.

When the mixtures are aged, by any of the two procedures, no significant differences are observed between the different types of mixtures. Therefore, the displacement at the maximum load is not enough sensitive to differentiate the behaviour of the aged mixtures due to the effect of the modifier.

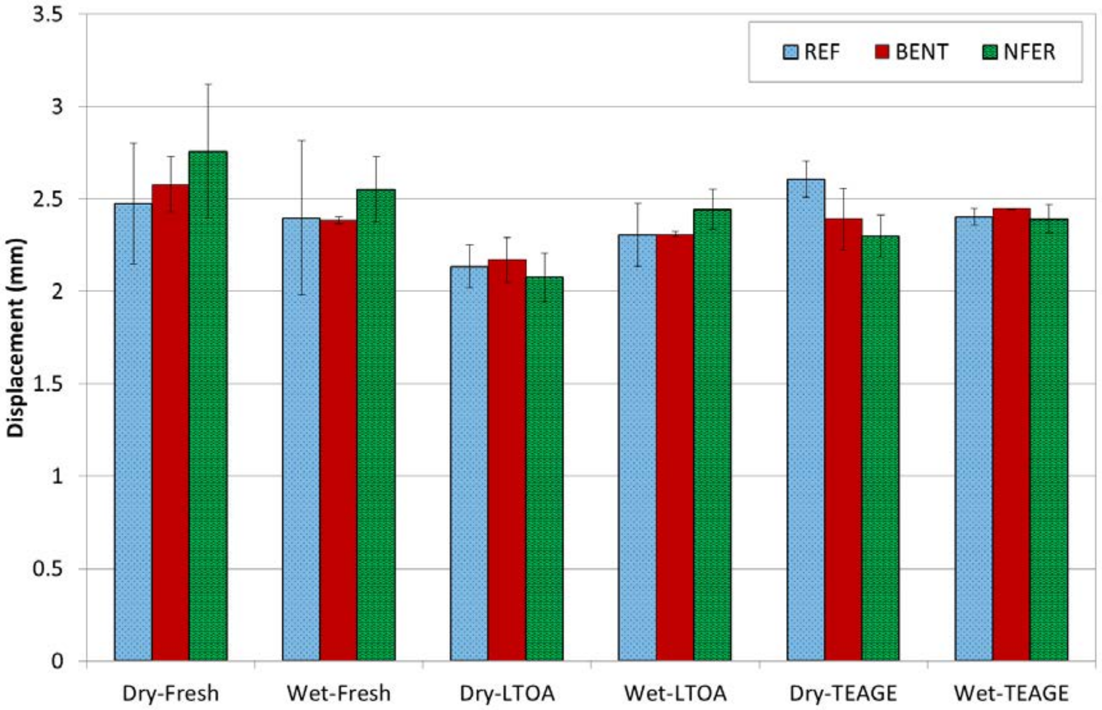

Figure 8. Average values of displacement when the peak load is reached in the indirect tensile strength test for the case studies.

Figure 9 shows the average values of the elastic energy for all the cases studied. The elastic energy is obtained as the area under the load-displacement curve until the peak value of the load is reached. Analysing this value together with the strength and the displacement values, a better idea of the behaviour of each type of mixture for the different conditions can be obtained. The elastic energy values for the dry fresh modified mixtures are greater than those for the reference mixture. In the case of nanoclay modified mixture, these differences come marked by high strength values, whereas for the nanoiron modified mixture, these differences come from higher values of displacement. Therefore, in this case where the displacement values are similar between the different mixtures, the elastic energy values follow the same trend than the strength values, which is observed in the wet fresh, aged by LTOA procedure conditions.

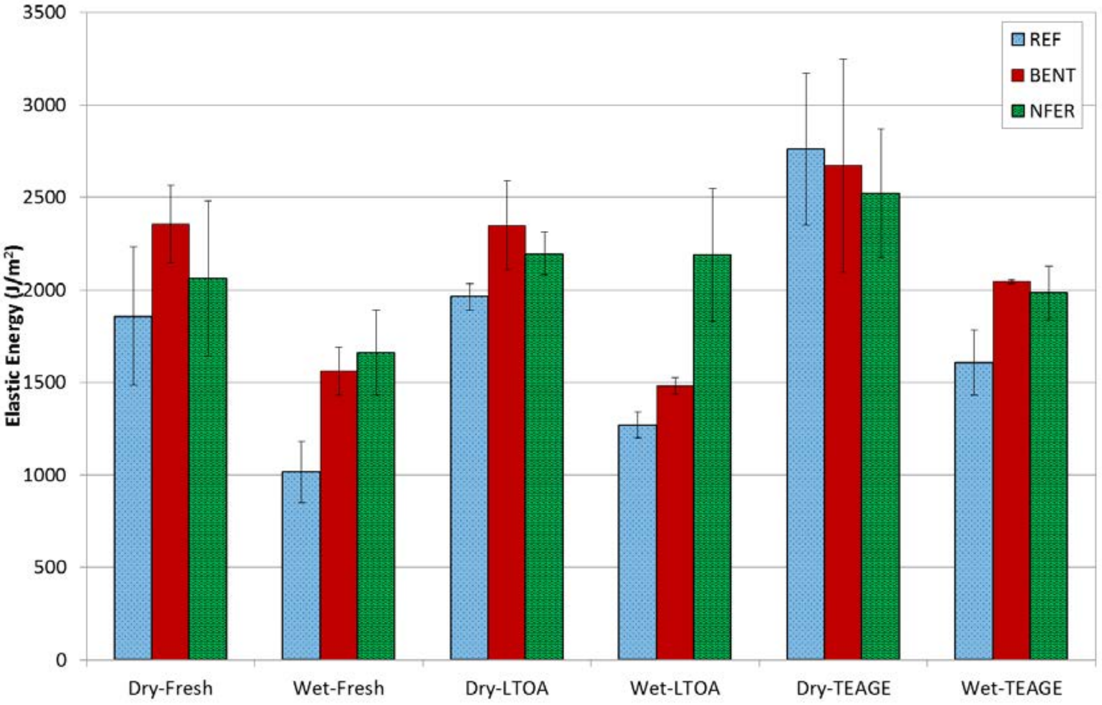

Figure 9. Average values of the elastic energy for the case studies.

Although from the results it is possible to conclude that ageing due to TEAGE is for sure not inducing similar ageing times to the one due to LTOA as it was described, and for sure more adapted of the region that is being simulated. Visual observation of the specimens aged by TEAGE procedure show signs of degradation in the form of visible alterations in its surface such as affected punctual spots and discoloration of the binder on the first mms of the samples, as it is expected once the ageing is made by UV action. Figure 10 shows an image of the specimens after being aged by the LTOA procedure and after being aged by the TEAGE procedure. A whiter colour is observed after TEAGE ageing. Figure 11 shows the punctual spots that have appeared in the samples of bituminous mixtures after being subjected to ageing.

Figure 10. Superficial differences between specimens aged by LTOA procedure (left) and specimens aged by TEAGE procedure (right).

Figure 11. Spots on the specimen after ageing by TEAGE procedure.

4. Conclusions

This study highlights the effect of the use of nanoparticles to modify the binder of asphalt mixtures, as well as the effect of ageing and moisture damage on the behaviour of these mixtures using the indirect tensile strength test. From this paper, a series of conclusions listed below have been obtained:

- For virgin samples (“fresh”) tensile strength of the mixtures increases by modifying the binder with nanoparticles, especially when the binder is modified with nanoclay.
- When the mixtures are subjected to moisture damage, their indirect tensile strength tends to decrease considerably. Analysing the values of the indirect tensile strength ratio, the nanoiron modified mixture shows the best behaviour against moisture damage.
- The values of the indirect tensile strength significantly increase with ageing due to the hardening of the binder, regardless of the ageing procedure used.
- Both ageing procedures, LTOA and TEAGE, have affected the samples behaviour and showed similar trends but in different degrees. This study has shown that it is worthy to try to match the ageing process (like TEAGE does) to the climate of the region where the mixtures are to be used.
- From the values of ageing index, nanoclay seems to retard the hardening process provoked by the ageing process but when both ageing and moisture damage are addressed, the nanoiron modified mixture probably deals better with hardening of the bitumen, increasing strength to a certain extent without loosing consistence and bond aggregate/binder properties, thus being more resistant to moisture aggression during the life cycle.
- The ageing by the TEAGE procedure seems to replicate better the climate effect on mixtures once has the possibility of adapting the ageing effect (by UV

incidence plus the simulation of the days with more than 5 mm of rain) on the mixtures to the number of years elected to analyse the behaviour for a certain region. Nevertheless, as stated in Crucho, et al. [15], some further validation works are needed to fully support the effectiveness of the procedure against others, namely LTOA.

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