Anti-aging additives: proposed evaluation process based on literature review

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ABSTRACT
Asphalt binder ageing alters its chemical and physical properties in a manner to make it brittle and more susceptible to thermal and fatigue cracking. The ageing rate of asphalt binders depends on a combination of different factors related to their chemical composition, environmental conditions and mix design. In order to prevent ageing, anti-ageing additives are used. The selection of the optimal additive should follow an iterative process. The additive choice is made concerning each case studied. Then, the selected modified asphalt binder is short-term aged by RTFOT and long-term aged by PAV and ultraviolet (UV) weathering. Rheology performance parameters are measured by DSR and BBR before and after ageing. Consequently, ageing indices are calculated to evaluate the appropriateness of the additive. The paper discusses several findings on the rheological level accordingly to the literature. Finally, a process is proposed to harmonise the research and the engineering practices in studying anti-ageing additives effects on asphalt binders.

ARTICLE HISTORY
Received 20 July 2020
Accepted 18 March 2021

KEYWORDS
Anti-ageing additives; asphalt binder; ageing; rheology; process

1. Introduction
Over millions of years, the maturation of natural products resulted in asphalt which is a very complex material due to its high content of different organic molecules (Petersen, 2009). The natural origin of asphalt is one of the reasons for its significant susceptibility to undergo ageing when used as a road construction material.

Asphalt binder ageing is a thermal-oxidative process commonly associated with three major mechanisms, namely volatilisation, steric hardening, and oxidation (Airey, 2003; Tauste et al., 2018). Volatilisation is the evaporation of lightweight components occurring during hot-mix asphalt (HMA) production (Hu et al., 2020). Steric hardening (physical ageing) refers to the progressive hardening of asphalt binder when there is the molecular restructuring of its molecules, which can be partially reversed by heat (Masson et al., 2005; Sirin et al., 2018). Oxidation is characterised by the chemical interaction between atmospheric oxygen, reactive oxygen species (ROS), and asphalt molecules which leads to irreversible changes (oxidative hardening) of the chemical and physical properties of the asphalt (Das et al., 2014; Hofko et al., 2020; Petersen & Glaser, 2011). The resistance of the asphalt binder
to oxidation depends on its component compatibility or state of dispersion of micellar components (Petersen, 2009).

Oxidative surface ageing is an irreversible chemical reaction between hydrocarbons of bitumen and available atmospheric oxygen. The ageing of asphalt binder results in the formation of more complex molecules with higher molecular weight and increased polarity (Lins et al., 2008; Lu & Isacsson, 2002; Ouyang et al., 2006a) as well as an increase in oxygen-containing functional groups through the reaction of the asphalt with atmospheric oxygen (Hofko et al., 2018; Lamontagne et al., 2001; Petersen, 2009). The combined action of several factors, such as aggregates characteristics (source, gradation, and absorption); air void content and distribution on asphalt mixtures; asphalt binder chemical composition; environmental and climatic conditions; the position of the asphalt mixture layer within the pavement structure; and asphalt mixing production process (the type of plant, silo storage conditions and the mixing temperature) have a significant impact on the ageing severity level (ageing rate) undergone by the asphalt binder (Farrar et al., 2013; Kandhal & Chakraborty, 1996; Kemp & Predoehl, 1981; Mogawer et al., 2012; Morian et al., 2011).

Given the wide variety of factors that can contribute to the ageing of asphalt binder, its simulation in the laboratory is not a trivial task, which explains the variety of available equipment to mimic it. Simplistically, the ageing process can be slip up into two phases: short-term and long-term (Kennedy et al., 1994). Short-term ageing happens during mixture production, transportation, laying and compaction. Long-term ageing, on the other hand, occurs during a pavement service life when it is exposed to environmental conditions and traffic loadings. Short-term and long-term ageing are replicated in the lab using different techniques. For instance, short-term ageing is usually achieved through a thin film oven test (TFOT) and mainly rolling thin film oven test (RTFOT). Long-term ageing is commonly simulated by pressure ageing vessel (PAV). To better simulate the field ageing, UV weathering can be employed to evaluate the effect of UV radiations on long-term ageing (Zhang et al., 2016). Also, the latest studies show that the addition of highly reactive ROS can bring the simulation of long-term ageing to a more realistic level (Hofko et al., 2017; Mirwald et al., 2020).

Ageing effects on asphalt binder could be evaluated by the characterisation of the changes of its physical and rheological properties. Physical tests include penetration tests, softening point tests, ductility tests, kinematic viscosity, extensibility tests, ductility tests, etc. Rheological evaluation of asphalt, whether modified or not, could be performed using a dynamic shear rheometer (DSR) and bending beam rheometer (BBR).

In addition, different chemical techniques have been considered to assess the effect of the ageing on chemical composition and microstructure of asphalt binder, among them atomic force microscopy (AFM), Fourier transform infrared (FTIR) spectroscopy, thin-layer chromatography, and high-pressure gel permeation chromatography (HP-GPC) and fluorescence spectroscopy and microscopy (Tauste et al., 2018).

The increase of brittleness of asphalt binder leads to the premature development of fatigue and thermal cracking, therefore, negatively affecting the durability of the asphalt pavements (Petersen, 2000; Prapaitrakul et al., 2009). Anti-ageing additives can be used to prevent the ageing of asphalt binders for pavement applications and improving the durability of asphalt pavements. These additives can have one or a combination of the following effects: free-radical scavenger (primary anti-oxidant), peroxide inhibitor (secondary anti-oxidant), radiation shield, oxygen shield, volatile fraction absorber, oxidation inhibitor and ultraviolet (UV) absorber (Tauste et al., 2018).

Upon the required effect, the additive can be used as an antioxidant, a polymer stabiliser, an organic warm-mix asphalt (WMA) additive, a synthetic WMA additive, asphalt modifier, a co-polymer enhanced with anti-oxidant agents, a filler and a UV antioxidant (Tauste et al., 2018). The selection of the appropriate additive needs to be looked at based on a case-by-case effort.

The approaches undertaken to assess the impact of using anti-ageing additives are manifold given the wide range of existing ageing protocols and the different tests performed on asphalt binders as well as the different climatic conditions around the world. Thus, the findings among the literature
are dispersed and sometimes conflicting. The benefits and the field of applications of each additive are not well captured. In addition, the pavement construction industry does not have sufficient evidence regarding the return of investment on such innovative products neither of its life-cycle cost-effectiveness (Glover et al., 2005).

Similarly, as for neat asphalt binder the ageing effects on asphalt binder by modified by anti-ageing additives could be evaluated using different techniques, but its physical outcome over time is the same, which is hardening and increased brittleness. This means that rheological tests are suitable for this objective since they detect hardening (increase in viscosity, increase in complex shear modulus, etc.), their theory is simple and easily understood by most researchers and practitioners, and the equipment is already available in most asphalt laboratories. Therefore, the authors recommend the use of rheological tests to assess the performance of anti-ageing additives under in-service temperature and UV exposure conditions.

Therefore, this paper aims to discuss the existing approaches of studying the influence of anti-ageing additives to come up with a harmonised process for investigating the appropriateness (technical and cost-effectiveness) of anti-ageing additives. This paper is organised as follows. First, the most relevant factors affecting the ageing mechanisms of asphalt binder as reported in the literature are discussed to define the inputs of the analysis. Then, a library of additives is provided as a guide to their selection in terms of type and content. After that, the ageing protocols and testing techniques are presented. Posteriorly, the anti-ageing additives performance evaluation process (based on rheological ageing indices) is proposed to assess the effectiveness of anti-ageing additives. Finally, it is presented a life cycle cost analysis to assess the economical feasibility of the use of anti-ageing additives in the construction of new asphalt pavements. This process is believed to harmonise the research and the engineering practices regarding the use of anti-ageing additives as asphalt binder modifier.

2. Factors affecting ageing mechanisms

The ageing phenomenon of modified or neat asphalt binders depends on several factors intrinsic to asphalt mixtures. The aggregate source, gradation and absorption have significant effects on ageing according to (Morian et al., 2011). Besides, Kandhal and Chakraborty (1996) have found a significant increase in ageing, which magnitude depends on the used asphalt binder type, for asphalt mixtures with an asphalt film thickness smaller than 9 µm. The ageing rate increases with an increase in air voids as found by Kemp and Predoehl (1981). Mogawer et al. (2012) reported that an increase of reclaimed asphalt pavement (RAP) content also increases the ageing rate. Thus, the components and their proportions in an asphalt mixture are factors that affect the ageing rate of asphalt binders.

Climatic conditions and the production process of the asphalt mixtures have significant effects on ageing. The type of plant (batch or drum), silo storage conditions, and the mixing temperature have a significant effect on short-term ageing as discussed by (Mogawer et al., 2012). Furthermore, the ageing of asphalt binders in a pavement structure depends on its depth. The deeper the position of the asphalt mixture layer within the pavement structure is, the least ageing occurs. However, asphalt ageing is not only limited to the upper 25 mm as it was tracked at a depth of 92 mm as reported by (Farrar et al., 2013). This effect could be combined with the void contents rate in the asphalt mixture (Petersen, 2009). Thus, aging phenomena need to be investigated in HMA application whether used for the wearing course or the base course. Long-term ageing is highly dependent on the in-service temperature (Sirin et al., 2018). (Rolt, 2000) determined that the exposure time is the dominant factor for ageing, which was confirmed by (Yu et al., 2009) under UV exposure. Nonetheless, the handling and the storage conditions of the asphalt binder contribute to the ageing intensity (Mercado et al., 2005).

In short, reported findings indicate that asphalt binder ageing is a complex phenomenon that is caused by many factors. As previously mentioned, mimicking the asphalt ageing phenomenon and evaluating its effect on the asphalt binder properties is not a trivial task, which becomes even
more challenging when anti-ageing additives are considered. Consequently, the authors recommend the use of rheological tests to assess the performance of anti-ageing additives under in-service temperature and UV exposure conditions.

3. Anti-aging additives library

The choice of type and the content of the anti-ageing additive added to the asphalt binder is an expert decision-making task, given the wide range of additives available and the appropriateness of each mix design (Apeagyei, 2011; Kassem et al., 2019).

Table 1 presents, in alphabetical order, the several additives and their combinations as reported in the literature. The upcoming sections of the paper will focus on the most efficient additives for each step of the ageing protocol as well as the performance testing.

4. Short-term ageing of asphalt binders

4.1. Short-term ageing protocols

During the production of asphalt mixtures, the asphalt binder undergoes short-term ageing due to the high temperatures involved during the mixing, transportation, laying, and compaction of the mixture (Isacsson & Zeng, 1998). Table 2 lists the main methods considered to simulate short-term ageing in asphalt binder modified with anti-ageing additives:

According to Table 2, short-term ageing of asphalt binders when anti-ageing additives are considered is simulated through the two main methods used to simulate the ageing of asphalt binders during the mixing production, known as RTFOT and TFOT.

Both tests are usually conducted at the same test temperature of 163°C. However, each procedure considers specific values of the binder film thickness, procedure duration, and agitation mechanisms. In the RTFOT test, the glass bottles with binder samples are arranged on a carousel subjected to constant rotation. The constant agitation and airflow supply during RTFOT prevent the formation of skin on the sample surface and assures the homogeneous oxidation of the binder. In addition to that, in the case of the modified asphalt binder, constant agitation can help to disperse the additive (Airey, 2003; Phromsorn & Kennedy, 1995; Speight, 2015). Regarding the TFOT, the film is not renewed, which enables the formation of the skin on the sample surface, limiting the loss of volatiles during ageing (Airey, 2003; Phromsorn & Kennedy, 1995).

The difference between both procedures affects the level of degradation that the binder will experience throughout the ageing simulation. (Shiau et al., 1992) and (Phromsorn & Kennedy, 1995) studies have shown that the degradation caused to binder by RTFOT is more severe than that caused by TFOT. The obtained result is expected since the configuration of the RTFOT allows a larger surface area of the binder to be subjected to constant oxidation when compared to TFOT.

Although most of the studies about anti-ageing additives modification considered RTFOT to assess to simulate the STA, there is some uncertainty about the effectiveness of the test configuration in simulating the ageing of modified asphalt binders. The main concern is that the addition of the modifier could significantly increase the viscosity of the binder, especially when fillers are considered, which would increase the thickness of the film formed during the RTFOT test and consequently, the severity of the ageing of the modified asphalt binder would be reduced (Airey, 2003; Lesueur et al., 2016).

For instance, some modified versions of the RTFOT test as the one proposed by (Bahia et al., 1998) sought to overcome the increase in the film due to the addition of the additive. For that, steel rods are inserted inside the glass bottles with the binder samples to improve the spreading of the binder and the formation of thin films (Airey, 2003). Besides, the consideration for heating of very thin films in the oven for an extended period instead of procedures in which the binder is rotated would be a plausible alternative for simulating the short-term ageing of modified asphalt binders with high viscosity (Lesueur et al., 2016).
Table 1. Studied anti-aging additives.

| Additive and additive content range (wt%) | Reference |
|------------------------------------------|-----------|
| (3-Aminopropyl)triethoxysilane (APTES) surface-modified silica nanoparticles (SNPs) (4%) | Karnati et al., 2019 |
| Bumetrizole (0.5–0.6%) | (Cong et al., 2012; Cong et al., 2013; Feng et al., 2016; Kuang et al., 2014) |
| Calprene 6120 (3%) | (Dessouky et al., 2015; Kassem et al., 2019) |
| Carbon black (0.5–5%) | (Cong et al., 2012; Cong et al., 2013; Kassem et al., 2019; Nare & Hlangothi, 2019) |
| Crumb rubber modifier (CRM) (8–16%) | (Ali et al., 2013; Ghavibazoo et al., 2015; Yin et al., 2013) |
| Crumb rubber (CR) treated with gama radiation or irradiated by microwave (5–15%) | (Ibrahim et al., 2015; Yin et al., 2013) |
| Diatomite (2–16%) | (Baldi-Sevilla et al., 2016; Cong et al., 2016; Zhang et al., 2019) |
| Dilaurylthiodipropionate (DLTDP) (1–1.5%) | (Apeagyei, 2011) |
| Furfural (1–5%) | (Apeagyei, 2011; Fini et al., 2016) |
| Hydrated lime (1–20%) | (Apeagyei, 2011; Kassem et al., 2019; Lesueur et al., 2016; Nare & Hlangothi, 2019) |
| Hydrophobic diatomite (4%; 8%; 12%; 16%) | (Cong et al., 2016) |
| Irganox E201 (3%) | (Dessouky et al., 2015) |
| Irganox 1010 (0.2–10%) | (Apeagyei, 2011; Kassem et al., 2019; Kuanget al., 2014; Nare & Hlangothi, 2019; Zhao et al., 2015) |
| Irganox 1076 (3%) | (Dessouky et al., 2015) |
| Layered double hydroxides (LDHs) (2–5%) | (Wu et al., 2012; Xu et al., 2015; Zhang et al., 2016; Zhao et al., 2015) |
| Lignin (2–10%) | (Arafat et al., 2019; Yu et al., 2017) |
| Limestone fillers (20%) | (Lesueur et al., 2016) |
| Micronized Polyethylene Terephthalate (PET) Waste (4–6%) | (Almeida e Silva et al., 2015) |
| Nanosilica (2–6%) | (Baldi-Sevilla et al., 2016; Fini et al., 2016; Karnati et al., 2019; Yao et al., 2013) |
| Nano calcium carbonate (CaCO3) (4%) | (Ouyang et al., 2006b; Yuan et al., 2019) |
| Nano silicon dioxide (SiO2) (4%) | (Cong et al., 2012; Cong et al., 2013; Feng et al., 2016; Kuang et al., 2014) |
| Nano titanium dioxide (TiO2) (4%) | (Rossi et al., 2018) |
| Naphthenoid oil (1%) | (Rossi et al., 2018) |
| Octabenzene (0.5–0.6%) | (Kassem et al., 2019; Zhao et al., 2015) |
| Organic intercalated layered double hydroxides (OLDHs) (3%) | (Xu et al., 2015) |
| Organo-montmorillonite (OMMT) (3%) | (Yu et al., 2009) |
| Phenol–formaldehyde resin (2%) | (Zaidullin et al., 2013) |
| Phenyl-β-naphthylamine (2%) | (Rossi et al., 2018) |
| Phospholipids (Commercial mix) (2%) | (Kassem et al., 2019) |
| Redicote AP (1%) | (Rossi et al., 2018) |
| Rice husk (2%) | (Dessouky et al., 2015; Kassem et al., 2019; Kuang et al., 2014) |
| Solprene 1205 (2–3%); Solprene 4318 (3%) | (Feng et al., 2016; Kuang et al., 2014) |
| Tinuvin 770 (0.6%) | (Rossi et al., 2018) |
| Triethoxyvinylsilane surface organic modified layered double hydroxides (TEVS-LDHs) (2%; 4%) | (Rossi et al., 2018) |
| Vitamin C (2%) | (Apeagyei, 2011; Dessouky et al., 2013; Kassem et al., 2019; Zhao et al., 2015) |
| Vitamin E (1–6%) | (Apeagyei, 2011; Dessouky et al., 2013; Kassem et al., 2019; Zhao et al., 2015) |
| Zinc dialkyldithiophosphate (ZDDP) (1%) | (Cong et al., 2012; Cong et al., 2013; Ouyang et al., 2006a, 2006b) |
| Zinc diethyldithiocarbamate (1–4%) | (Haghshenas et al., 2019) |
| Carbon black (5%) + Dilaurylthiodipropionate (DLTDP) (1.5%) | (Apeagyei, 2011) |
| Carbon black (3%) + Irganox 1010 (3%) | (Cong et al., 2012; Cong et al., 2013) |
| Carbon black (3%) + Vitamin E (3%) | (Apeagyei, 2011) |
| Carbon black (1%) + Octabenzene (0.5%) | (Cong et al., 2012; Cong et al., 2013) |
| Carbon black (1%) + Bumetrizole (0.5%) | (Apeagyei, 2011) |
| Furfural (2%) + Vitamin E (2%) | (Cong et al., 2012; Cong et al., 2013) |
| Furfural (1.5%) + DLTDP (2%) | (Apeagyei, 2011) |
| Irganox 1010 (1%) + Dilaurylthiodipropionate (DLTDP) (1%) | (Apeagyei, 2011) |

(continued).
Table 1. Continued.

| Additive and additive content range (wt%) | Reference |
|------------------------------------------|-----------|
| Irganox 1010 (0.6%) + Layered double hydroxides (LDHs) (3%) | (Zhao et al., 2015) |
| Irganox 1010 (3%) + Vitamin E (3%) | (Apeagyei, 2011) |
| Irganox 1010 (2%) + Irgafos P-EPQ (3%) | (Kassem et al., 2019) |
| Vitamin E (1–2%) + Irgafos P-EPQ (4–9%) | (Cong et al., 2012; Cong et al., 2013) |
| Vitamin E (2–3%) + Hydrated lime (2%) | (Zhao et al., 2015) |
| Zinc dialkyldithiophosphate (ZDDP) (1%) + Bumetrizole (0.5%) | (Apeagyei, 2011) |
| Zinc dialkyldithiophosphate (ZDDP) (1%) + Octabenzone (0.5%) | (Kassem et al., 2019) |

4.2. Short-term aged modified asphalt properties

Rutting (permanent deformation) is one of the major structural distresses found in asphalt pavements. It is related to depression in the wheel path on the surface of the pavement which results in a reduction in the pavement service life, an increase of costs with early rehabilitation of pavement, and, the reduction in the safety and comfort of the road users (Azari & Mohseni, 2013; Khan et al., 2013; Sun, 2016).

This type of distress mainly occurs during the early life of asphalt pavements and is significantly affected by factors such as temperature and loading (Dessouky et al., 2013; Singh et al., 2017). For this reason, the performance tests usually consider unaged and short-term aged samples to predict the rutting performance of asphalt pavements. The performance can be evaluated using chemical, physical, and rheological methodologies. However, as previously discussed, only rheological methods are addressed in this research work.

The Superpave rutting parameter, $G^*/\sin\delta$, is one of the most widely used parameters to assess the rutting potential of asphalt binders (Kennedy et al., 1994). The higher this parameter is the better the rutting resistance of the material is (Das & Singh, 2019). Several authors predicted the ageing susceptibility of short-term aged asphalt binder based on the ageing index ($A_{STA}$) (Equation 1) calculated considering the Superpave rutting parameters of unaged and short-term aged asphalt binder.

$$A_{STA} = \frac{G^*/\sin\delta_{after~short~term~aging}}{G^*/\sin\delta_{unaged}}$$

A value of the ageing index higher than 1 indicates that the material is already aged (Arafat et al., 2019). Table 3 shows the effect of anti-ageing additives on the ageing index of neat asphalt and modified asphalt binders. In this study, only the cases where the addition of anti-ageing additive resulted in 20% or higher reduction in the $A_{STA}$ are reported. The shaded grey values shown in this table were calculated based on published data or curves given in the cited publications while the other values are reported directly from their references.

For a given neat asphalt binder, it was observed that in general, the modification resulted in a reduction in the ageing index, which shows the potential of the studied anti-ageing additives in reducing short-term ageing. Among the additives that most contributed to increasing the resistance of the neat binder against short-term ageing, it is highlighted the compound of DLTDP (2.0 wt%) and Furfural (1.50 wt%); Furfural (2.0 wt%); crumb rubber modifier (8.0 wt%); and the compound of styrene-butadiene rubber (3.0 wt%), weathered coal (3.0 wt%) and carbon black (2.0 wt%).

Given the above, it is possible to verify that most of the studied additives showed effectiveness in increasing the resistance of the asphalt binder to short-term ageing. However, this improvement depends on the type of asphalt binder and the amount of additive added.
Table 2. Reported testing parameters used to short-term aging of modified binders.

| Aging method | Standard | duration (minutes) | T. (°C) | Reference |
|--------------|----------|--------------------|---------|-----------|
| TFOT         | ASTM D1754 (ASTM, 2002) | 300                | 163     | (Cong et al., 2013; Feng et al., 2016; Ouyang et al., 2006b; Xu et al., 2015; Yu et al., 2009; Zhang et al., 2016; Zhang et al., 2009; Zhao et al., 2015) |
| RTFOT        | ASTM D2872 (ASTM, 2012) | 85                 |         | (Ali et al., 2013; Cong et al., 2012; Dessouky et al., 2013; Fini et al., 2016; Haghshenas et al., 2019; Karnati et al., 2019; Almeida e Silva et al., 2015; Yuan et al., 2019; Zhang et al., 2009) |
| RTFOT        | ASTM D2872 (ASTM, 2012) |                    | 163; 170; 180; 190 | (Ghavibazoo et al., 2015) |
| RTFOT        | AASHTO T240 (AASHTO, 2013) |                    | 163     | (Apeagyei, 2011; Arafat et al., 2019; Kassem et al., 2019; Nazari et al., 2018; Xu et al., 2017) |
| RTFOT        | NR       | NR                 | 163; 143 | (Banerjee et al., 2012) |
| RTFOT        | NR       | 85                 | 163     | (Ibrahim et al., 2015) |
| RTFOT        | EN 12607 (CEN, 2007)    | 75                 | 163     | (Rossi et al., 2018) |
| RTFOT        | NR       |                    |         | (Nare & Hlangothi, 2019) |

Note: NR: not reported.
### Table 3. Aging index after short-term aging.

| Additive and additive content (wt%)                  | Asphalt binder | T. (°C) | AI<sub>STA</sub> | Reference                  |
|--------------------------------------------------|----------------|---------|-----------------|---------------------------|
| Neat asphalt binder                               | AH – 90        | 60      | 2.12            | (Zhanget al., 2009)       |
| weathered coal (6.0%) + Carbon black (2.0%)       |                |         | 1.63            |                           |
| styrene butadiene rubber (3.0%) + weathered coal (3.0%) + Carbon black (2.0%) | | | 1.37 | |
| Neat asphalt binder                               | PG 64–22       | 64      | 2.32            | (Apeagyei, 2011)          |
| Carbon black (5.0%) + DLTDP (1.5%)                |                |         | 1.73            |                           |
| Furfural (2.0%) + Vitamin E (2.0%)                 |                |         | 1.72            |                           |
| Carbon black (3.0%) + Irganox 1010 (3.0%)         |                |         | 1.61            |                           |
| DLTDP (2.0%) + Furfural (1.5%)                    |                |         | 1.44            |                           |
| Furfural (2.0%)                                   |                |         | 1.46            |                           |
| Neat asphalt binder                               | 80/100 pen     | 76      | 1.80            | (Ali et al., 2013)        |
| crumb rubber modifier (8.0%)                      |                |         | 1.20            |                           |
| Neat asphalt binder                               | PG 64–22       | 76      | 8.78            | (Dessouky et al., 2013)   |
| styrene-ethylene-butadiene-styrene (3.0%)         |                |         | 1.79            |                           |
| Hindered phenols (HP 2) (3.0%)                    |                |         | 2.49            |                           |
| Neat asphalt binder                               | PG 76–22 (SBS) | 82      | 2.05            | (Xu et al., 2017)         |
| Lignin (5.0%)                                     |                |         | 1.58            |                           |
| Lignin (10.0%)                                    |                |         | 1.60            |                           |

5. **Long-term ageing of asphalt binder**

5.1. **Long-term ageing protocols**

The long-term ageing of the asphalt pavement occurs during its service life. It is related to the interaction of the pavement with external factors, such as temperature, light (i.e. ultraviolet radiation), reactive gases, and water-soluble reactants, which are found in the troposphere (Hofko et al., 2015).

Given the variety of factors that can interfere with ageing, simulating this phenomenon in the laboratory is challenging and difficult to achieve. Because of this, long-term laboratory ageing procedures generally only consider the effect of some of those parameters on asphalt binder ageing.

The PAV test is the most commonly used ageing method to simulate long-term ageing of the asphalt binder during the pavement service life, which is required in the Superpave process. However, the test parameters such as temperature and pressure are quite different from those found in the field. For instance, the study of Al-Azri et al. (2006) showed that PAV was not adequate to simulate field ageing. One of the possible explanations for such findings is the fact that the microstructure of the asphalt binder is disrupted because of the high temperature (Petersen, 2009).

Also, the oxidative ageing effect simulated during PAV is different from that observed in photochemical ageing due to the action of UV radiation (Airey, 2003). A possible solution to improve the ageing simulation in the laboratory is the development of ageing devices that combine the effect of oxidative gases, temperature, and UV radiation to mimic the asphalt ageing process in the field (Airey, 2003; Mirwald et al., 2020).

The PAV and UV ageing procedures were considered by Feng et al. (2016), to assess the effectiveness of three different UV absorbers to protect the asphalt binder against ageing. The FTIR spectra of binders after both types of ageing showed that the severity of ageing caused by PAV is greater than that observed for the UV aging conducted in the study.

However, it is not possible to state that the ageing simulated by PAV ageing is always more severe than UV ageing since there is a lack of standardisation concerning the inputs (UV intensity, film thickness, and test duration) considered in UV aging procedures.

The study of Kuang et al. (2014) indicates that the ageing severity undergone by the asphalt binder depends on the UV intensity, the origin of the asphalt binder and the type of anti-ageing additive used.
Table 4. Reported PAV parameters used in long-term aging of modified binders.

| Standard                  | duration (hours) | T. (°C) | Pressure (MPa) | References                                      |
|---------------------------|------------------|---------|----------------|------------------------------------------------|
| ASTM D 6521 (ASTM, 2013) | 20               | 100     | 2.1            | (Baldi-Sevilla et al., 2016; Dessouky et al., 2013; Feng et al., 2016; Fini et al., 2016; Karnati et al., 2019; Yu et al., 2009) |
| ASTM D 6521 (ASTM, 2013) | 20; 40; 80       |         |                | (Haghshenas et al., 2019)                       |
| EN 14769 (CEN, 2012)     | 25               |         |                | (Lesueur et al., 2016)                          |
| AASHTO R28 (AASHTO, 2012)| 20               |         |                | (Arafat et al., 2019; Kassem et al., 2019; Nazari et al., 2018; Xu et al., 2017) |

Table 5. Reported UV parameters used in long-term aging of modified binders.

| Power (W) | UV wavelength (nm) | UV intensity (μW/cm²) | Film thickness (mm) | T. (°C) | Time (days) | References |
|-----------|--------------------|-----------------------|---------------------|---------|-------------|------------|
| 500       | 340                | NR                    | 2                   | 80      | 0–18        | (Yu et al., 2009) |
| 500       | 340                | 450,000               | 3                   | 60      | 7           | (Cong et al., 2012) |
| NR        | NR                 | 15,000                | 1.25                | 50      | 6           | (Wu et al., 2012)  |
| 500       | 340                | 450,000               | 3                   | 60      | 7           | (Cong et al., 2013) |
| 500       | 365                | 950; 1200             | 3                   | 60      | 7           | (Kuang et al., 2014) |
| NR        | 365                | 1200                  | NR                  | 60      | 9           | (Xu et al., 2015)  |
| 500       | NR                 | 1200                  | NR                  | 60      | 9           | (Zhao et al., 2015) |
| 500       | NR                 | 1200                  | 2                   | 60      | 6           | (Feng et al., 2016) |
| 500       | 365                | 2000                  | NR                  | 60      | 7           | (Zhang et al., 2016) |

Note: NR: not reported.

In addition, the study of Zeng et al. (2018) found that the exposure time also has a significant effect on the penetration and diffusibility of UV radiation on the asphalt binder sample.

Table 4 presents the standards and parameters considered for the oxidative ageing of the binders according to the PAV procedure, while Table 5 shows the inputs considered by different studies to simulate the ageing caused by the action of UV radiation.

According to Table 4, most studies using PAV to simulate the long-term ageing of asphalt binder modified with anti-ageing additives consider the same values for temperature, pressure, and time duration. Except (Lesueur et al., 2016) and (Haghshenas et al., 2019) in which the duration of PAV procedure has been increased. The study of (Haghshenas et al., 2019) considered a long duration to evaluate the feasibility of the use of an anti-ageing additive (zinc diethyldithiocarbamate) in the construction of longer-lasting pavements (20 years). The results of rheological and chemical characterisation of aged modified binder showed the potential of zinc diethyldithiocarbamate to increase at twice the service life of the pavement.

Table 5 indicates that the parameters and practices considered to simulate the photo-degradative effect of ultraviolet radiation on the ageing of asphalt binders are quite different from those adopted in the PAV test. The main differences are temperature, UV intensity, and test time considered by each study.

Due to the complexity and variety of additives with different active principles to minimise the long-term ageing of asphalt binders, it is essential to improve the long-term ageing procedures to better predict the effectiveness of these additives in the field. Furthermore, the impact of the two different types of parameters (thermal oxidation in the PAV and UV radiation) needs to be considered in regards to the chemical class of the additive. While organic additives might be prone to decompose upon exposure to UV radiation, inorganic additives could behave differently. Besides, it is worthy to note that the free radicals additives are more effective against photo-oxidation than dark oxidation (Petersen, 2009). Additionally, other factors like reactive oxygen species (Mirwald et al., 2020), moisture, and UV seem to be important to achieve a realistic, yet efficient long-term ageing simulation.
### Table 6. Published fatigue cracking performance after long-term aging of anti-ageing additives.

| Additive and additive content (wt%) | Asphalt | Aging protocol | T (°C) | AILTA  | PI    | Reference                   |
|------------------------------------|---------|----------------|--------|--------|-------|-----------------------------|
| Neat asphalt binder                | PG 64–22 | RTFO + PAV     | 25     | 4.98   | –     | (Apeagyei, 2011)            |
| DLTPD (2%) + Furfural (1.5%)       |         |                |        | 3.34   | 0.56  |                             |
| DLTPD (1.5%)                       |         |                |        | –      | 0.62  |                             |
| Neat asphalt binder                | Pen grade 70 | TFOT + PAV | NR     | 5.98   | –     | (Zhao et al., 2015)         |
| LDH (0.6%)                         |         |                |        | 3.18   | 0.84  |                             |
| Irganox 1010 (3%)                  |         |                |        | 2.64   | 0.64  |                             |
| LDH (0.6%) + Irganox (3%)          |         |                |        | 3.32   | 0.46  |                             |
| Neat asphalt binder                | PG 64–22 | RTFO + PAV     | 25     | 4.47   | –     | (Kassem et al., 2019)       |
| Hydrated lime (2%)                 |         |                |        | 3.12   | 0.77  |                             |
| Redicote AP (1%)                   |         |                |        | 3.53   | 0.62  |                             |
| Neat asphalt binder                | PG 67–22 |                |        | 5.64   | –     |                             |
| Irganox 1010 (1%)                  |         |                |        | 3.97   | 0.69  |                             |
| Redicote AP (1%)                   |         |                |        | 3.07   | 0.54  |                             |
| Furfural (2%) + HCl                |         |                |        | 4.5    | 0.59  |                             |

#### 5.2. Long-term aged modified asphalt properties

Modified asphalt binders exposed to long-term ageing should have adequate performances against fatigue and low temperature cracking according to (Kennedy et al., 1994). For instance, the complex modulus $G^*$ is increased at high temperatures and decreased at low temperatures with the addition of polymer additives (Ruan et al., 2003).

Resistance to fatigue cracking of a long-term aged modified asphalt binder is evaluated using its $G^* \times \sin \delta$ value, which is obtained from a DSR test according to (ASTM, 2008b) at intermediate temperatures (Kennedy et al., 1994). This parameter $(G^* \times \sin \delta)$ assesses the brittleness of asphalt after ageing. Even though, the standard calls for one testing temperature and frequency, several researchers performed temperature and frequency sweep tests to investigate additives influence on performance. For instance, (Wu et al., 2012) established master curves of the complex modulus $G^*$ and phase angle $\delta$ and showed that neat asphalt binder has the highest $G^*$ and the lowest $\delta$ for all testing temperatures as compared to two modified asphalt binder after six days of UV weathering.

To quantify the performance of modified asphalts, an index based on stability after ageing, ageing index (AILTA), has been proposed as shown by Equation (2). Also, Dessouky et al. (2015) adopted a performance index (PI) as shown by Equation (3). A decrease in AI indicates better stability of the aged asphalt blend. Besides, the lower the PI the better resistance to fatigue cracking the blend is since it becomes less brittle.

A summary of the evaluation of fatigue performances for some tested additives can be found in Table 6. In this study, only the cases where the addition of anti-ageing additive resulted in a 20% or higher reduction in the AILTA are reported. The shaded grey values shown in this table were calculated based on published data or curves given in the cited publications while the other values are reported directly from their references. The calculated and reported AILTA values to vary between 2.33 and 5.5 with an average of 3.12. Similar to the short-term ageing approach, only the cases where the antioxidant effect of the additive was significant were presented. For all researched additives, the AILTA was reduced as compared to that found for neat asphalt.

For the PI values, an interval from 0.46 (best performing additive tested by Zhao et al. (2015)) to 1.59 (worst performing additive tested by Dessouky et al. (2015)) was found with an average of 0.75 (a minimum of 25% gain in fatigue performance). The fatigue cracking resistances is enhanced in almost all the studied cases.

$$A_{ILTA} = \frac{G^* \sin\delta_{after \ long \ term \ aging}}{G^* \sin\delta_{unaged}},$$ (2)
\[ PI = \frac{G^* \sin \delta_{\text{aged modified asphalt}}}{G^* \sin \delta_{\text{aged neat asphalt}}} \]  

(3)

Resistance to low-temperature cracking of a binder is determined using the Bending Beam Rheometer (BBR) test according to standard (ASTM, 2008a). In this test, the stiffness \( S \) and the \( m \) value (rate of change of the logarithm of stiffness concerning the logarithm of time) are measured after 60 s of loading. Two indices could be calculated from tested specimens after long-term ageing to capture the effects of additives on asphalt binder’s low-temperature behaviour. The stiffness index (SI), which is based on measured stiffness as shown by Equation (4), and the \( m \)-value index (MI), which is based on calculated \( m \) values as shown by Equation (5). A decrease (increase) of the SI (MI) indicates a better resistance to low temperature cracking. Several BBR test results from the literature are reported in Table 7. The values shaded in grey are calculated from data or curves given in the cited references. The majority of researched additives decrease the low-temperature resistance of asphalt binder except those tested by Apeagyei (2011) and Dessouky et al. (2015), which improved low-temperature performance.

\[ SI = \frac{S_{\text{Modified asphalt}}}{S_{\text{Neat asphalt}}} \]  

(4)

\[ MI = \frac{m_{\text{Modified asphalt}}}{m_{\text{Neat asphalt}}} \]  

(5)

6. Utilisation of Fourier transform infrared (FTIR) spectroscopy to evaluate short-term and long-term aging indices of modified asphalt binders

Due to the incorporation of oxygen during asphalt binder ageing, functional groups like sulfoxides or carbonyls are formed. The formation of these groups is influenced by the ageing temperature and can be correlated to the increase in viscosity, which leads to the binder hardening due to its oxidation (Herrington et al., 1994; Petersen & Glaser, 2011).

Fourier transform infrared spectroscopy (FTIR) has been used extensively to track ageing in asphalt binders. This technique allows the identification and measurement of IR active functional groups that are present within the material (Karlsson & Isacsson, 2003; Lamontagne et al., 2001; Lu & Isacsson, 1998; Martin et al., 1990). Carbonyl and sulfoxide indices are generally calculated based on the absorbance peaks in the band regions 1700 cm\(^{-1}\) and 1030 cm\(^{-1}\), respectively.

In the case of asphalt binders modified with anti-ageing additives, it was found that the sulfoxide index can lead to erroneous conclusions about the oxidative ageing of the binder (Ouyang et al., 2006a; Xu et al., 2017; Yao et al., 2013). For this reason, in this study, it is recommended the cautious use of carbonyl and sulfoxide indices to evaluate the effect of the additive on the ageing resistance of asphalt binder. Since the addition of the specific additive may dissemble the increase of such compounds on the aged binder leading the erroneous interpretations.

7. Proposed anti-ageing additives performance evaluation process

Based on the literature review, a flowchart was developed that exhibits a process to assess anti-ageing additives. The additive selection is an expert choice, which can be based on the above-discussed findings. The WMA additives do not have any adverse effect on ageing of the asphalt binder (Liu & Glover, 2015). Besides, this process extends the investigation to the asphalt mastic and mixture level. VAPro (Steiner et al., 2016) and DSR tests on asphalt mastic (Hospodka et al., 2018) are suggested for this aim. At the end of each step, evaluation indices are calculated to highlight whether the additive is worthy or not from an ageing resistance perspective. Finally, the profitability of the additive will be evaluated in an efficient economic tool such as the life cycle cost analysis (LCCA). This process may overcome the gap between academia and the industry by initiating a comprehensive engineering process.
| Additive and additive content | Asphalt binder | Aging protocol | $T$ (°C) | SI | MI | Reference |
|-------------------------------|----------------|----------------|---------|----|----|-----------|
| FT-paraffin Sasobit (3%)     | Pen grade 170  | RTFO + PAV     | −25     | 1.14 | –  | (Edwards et al., 2005) |
|                              | Pen grade 180  |                |         | 1.09 |    |           |
|                              | Pen grade 210  |                |         | 1.08 |    |           |
| Montan wax (3%)              | Pen grade 170  |                | −25     | 0.96 |    |           |
|                              | Pen grade 180  |                |         | 1.07 |    |           |
| Polyethylene wax (3%)        | Pen grade 170  |                | −25     | 0.95 |    |           |
|                              | Pen grade 180  |                |         | 0.87 |    |           |
| DLTDP (2%) + Furfural (1.5%) | PG 64–22       | RTFO + PAV     | −12     | 0.68 | 1.06| (Apeagyei, 2011) |
| ZDDP (1%) + UV351 (0.5%)     | Pen grade 60/80 | TFOT + UV weathering | −12/−18 | 2.26/1.1 | 1.27/0.97 | (Cong et al., 2013) |
| ZDDP (1%) + UV326 (0.5%)     | Pen grade 60/80 | TFOT + UV weathering | −12/−18 | 0.86/0.91 | 1.40/0.97 |           |
| Carbon Black (1%) + UV351 (0.5%) | PG 58–34   | RTFO + PAV     | −24     | 1.31 | 0.97| (Yao et al., 2013) |
| Carbon Black (1%) + UV326 (0.5%) | PG 58–34   | RTFO + PAV     | −24     | 1.42 | 0.95|           |
| Nano silica (4%)             | PG 64–22      | RTFO + PAV     | −12     | 1.2 | 0.95|           |
| Carbon Black (1%) + UV326 (0.5%) | PG 64–22   | RTFO + PAV     | −12     | 1.34 | 0.95|           |
| Calprene 6120 (3%)           | PG 64–22      | RTFO + PAV     | −12     | 1.22 | 0.95|           |
| Wood Lignin (10%)            | PG 64–22      | RTFO + PAV     | −12/−18 | 1.33/1.18 | 0.95/0.97 | (Xu et al., 2015) |
| MORLIEM 5000 (0.5%) + CRM (10%) | PG 64–22 | RTFO + PAV | −6/−12/−18 | 0.83/0.84/0.9 | 0.98/0.99/0.95 | (Tang et al., 2019) |
| EVOTHERM M1 (0.5%) + CRM (10%) | PG 64–22 | RTFO + PAV | −6/−12/−18 | 0.83/0.88/0.89 | 0.96/0.94/0.95 |           |
| AD-here LOF-65-00 (0.5%) + CRM (10%) | PG 64–22 | RTFO + PAV | −6/−12/−18 | 0.71/0.8/0.74 | 0.95/0.92/0.9 |           |
| Tourmaline powder of 325 meshes (18%) | Pen grade 70 | RTFO + PAV | −12/−18 | 1.26/1.32 | 0.96/0.94 | (Ye et al., 2020) |
| Tourmaline powder of 3000 meshes (18%) | Pen grade 70 | RTFO + PAV | −12/−18 | 1.39/1.42 | 0.98/0.88 |           |
| Negative ions treated tourmaline powder of 325 meshes (18%) | Pen grade 70 | RTFO + PAV | −12     | 1.09 | 0.99|           |
|                              |                |                | −18     | 1.19 | 0.97|           |
Table 8. The pavement structure.

| Layer          | Material        | Thickness (mm) | Modulus (MPa) |
|----------------|-----------------|----------------|---------------|
| Wearing layer  | HMA             | 50             | Variable      |
| Base layer     | Crushed stone   | 200            | 360           |
| Sub base layer | Untreated material | 200        | 150           |
| Subgrade       | Soil            | –              | 75            |

8. Life cycle cost analysis (LCCA)

LCCA is an economic analysis tool, which can reveal whether or not a technically sound optimal alternative could not be optimal concerning expenditure aspects (Loulizi et al., 2019). It is based on an analytical technique taking into account costs relevant to the sponsoring agency, owner, facility operator, and roadway user for a given analysis period. These costs are usually initial investment cost, rehabilitation and maintenance cost, and user cost (time delay in a work zone). The salvage value (how much is the asset is worth at the end of the analysis period) of the project is also considered in the LCCA. Finally, the net present value (NPV) and/or the equivalent uniform annual cost (EUAC) of the whole project could be used to evaluate the cost-effectiveness of the additive (Bull, 2015).

To illustrate the concept, a case study is presented, which considers a hypothetical pavement section consisting of the layers shown in Table 8.

The analysis period was taken to be 20 years and a 3% discount rate was selected. Two alternatives were considered with a wearing surface made using neat asphalt for alternative one while alternative two considers the same wearing surface mix, but using an anti-aging additive. The performance results \((G^{\ast} \text{ and the phase angle } \delta)\) of both types of asphalt were taken from the reference by Fini et al. (2016). The complex modulus of mix was calculated using Witzack Equation 1-40D (Bari & Witczak, 2006). The average annual daily traffic (AADT) is taken equal to 1000 vehicles in which 4% are trucks and the growth rate is assumed as 3%. The French mechanistic-empirical method (AFNOR, 2019) was then used with both structures to determine the pavement service lives. All needed unit costs for the analysis were adopted from the Idaho State Department of Transportation (USDA, 2017). Finally, RealCost 2.5 software (FHWA, 2016) was used to perform the LCCA and calculate the EUAC for both alternatives. Figure 2 shows the expenditure stream for both alternatives with their respective salvage values at the end of the 20-year analysis period. The EUAC for agency costs were found to be $16770 and $16690 for alternatives 1 and 2, respectively. While the user costs EUAC were found to be $710 and $600 for alternatives 1 and 2, respectively. Thus, Alternative 2 with the anti-ageing additive, results in a reduction in both agency and user costs, which means that the used anti-aging additive has a valuable economic benefit.

9. Conclusions

Anti-ageing additives can improve the ageing resistance of the asphalt when used in HMA or WMA. This statement has been discussed by several researchers over the years. This paper emphasis the following outcomes based on evidence found in the literature review:

(i) The ageing of the asphalt is dependent on the asphalt mixture design, the in-service temperature, the exposure to atmospheric factors (UV, moisture, ROS) and its position in the pavement structure.

(ii) Short-term ageing simulation of asphalt binders modified with anti-ageing in the lab is mainly carried out by TFOT and RTFOT. Based on the literature, it can be concluded that the RTFOT is an efficient and realistic short-term ageing protocol to mimic the ageing process during asphalt mix production in the laboratory. However, it is recommend to consider modified versions of RTFOT test when the viscosity of the asphalt binder is significantly increased by the anti-ageing additive.
(iii) DSR tests are performed on short-term aged asphalt to evaluate the rutting parameter. The ratio of the aged value by the unaged value of this parameter is a common metric of ageing. The resistance to short-term ageing is improved by almost all the anti-ageing additive.

(iv) Long-term ageing simulation in the lab is usually conducted using the PAV. Since its parameters do not reflect the conditions found in the field, various approaches to realistically simulate binder ageing in the lab were implemented by the addition of UV radiation. As this reflects only one
of many parameters from the field, further investigations are needed to fully assess realistic, yet
time-efficient binder ageing in the laboratory.

(v) The fatigue parameter is determined by DSR tests after long term-ageing. Besides, BBR is per-
formed at this phase to assess low temperature cracking performance. Indices such as PI, SI, and
MI are suitable to assess the performance improvement due to the used additive. Fatigue performance
is often improved. However, low-temperature cracking resistance is commonly affected
negatively.

(vi) FTIR spectroscopy is a great tool to study the changes in the materials chemical composition due
to ageing. However, in the case of anti-ageing additives, the application of FTIR spectroscopy
needs to be treated with care, as their respective signals may interfere with the signals from
carbonyls and sulfoxides, which are used to determine ageing.

This paper brings up a process that could harmonise all the effort to assess the benefits of the anti-
ageing additives. This process is comprehensive and based on actual engineering practices. Currently,
the approach is purely based on rheological assessment. Since other physio-chemical analysis meth-
ods are more commonly used in recent years, a more comprehensive assessment including suitable
microscopic and spectroscopic methods will improve our understanding of the complex mechanisms
linked to asphalt ageing.

Acknowledgments
This study is part of the SAFERUPI Project, an innovative training network devoted to develop ‘safe, accessible, and
urban pavements’. The authors acknowledge TU Wien Bibliothek for financial support through its Open Access Funding
Programme.

Disclosure statement
No potential conflict of interest was reported by the author(s).

Funding
This work was supported by H2020 Marie Sklodowska-Curie Actions [grant number 765057].
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