Use of Zinc Oxide in Asphalts

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Zinc oxide (ZnO) is a wide-gap semiconducting material which is chemically stable at high temperatures and has been shown to be compatible with asphalt binders. Additionally, semiconducting nanoparticles such as ZnO could help to improve urban air quality. This has encouraged the use of this material as a binder and asphalt mix modifier.

Keywords: asphalt ; binder ; modifier ; zinc oxide (ZnO) ; nano-ZnO

1. Introduction

Zinc is the fourth-most widely used metal in the world after iron, aluminum, and copper [1]. Zinc oxide (ZnO) is the main chemical product produced from metallic zinc. Nano-zinc oxide (nano-ZnO) is produced from the synthesis process of an aqueous solution of zinc nitrate ($ZnNO_3$) and hexamine ($C_6H_{12}N_4$) with some reagents such as polyethylene glycol at a temperature above 90 °C [2]. ZnO powder has traditionally been used as a white pigment and as an additive for rubber. Its use is estimated to be between 50% and 60% in the rubber industry (as a vulcanization activator and improving thermal conductivity). The second-largest application is in ceramics [3]. This is mainly because ZnO is a material with high heat capacity, high conductivity, and thermal and chemical stability [4]. It is also a material with a low coefficient of expansion and optical ultraviolet (UV) absorption [5]. In the concrete industry, its addition in small quantities to Portland cement can delay setting and hardening [6]. It can also improve the water resistance of concrete [7]. ZnO is also used in plastics, pigments, and coatings; in cosmetic, medical, and dental applications; in fertilizers, animal feed, and dietary supplements; and in electronics, optoelectronics, glass, and lubricants, etc. [5][8][9][10].

Zinc oxide (ZnO) is a wide-band gap semiconductor material with piezoelectric properties [11][12]. These properties, in conjunction with its nanometer size, have attracted the interest of numerous researchers in the field of modified asphalt to improve UV aging resistance [2][13][14][15][16][17]. It is a high-refractive-index material, as it can be an electrical conductor when properly doped, and is thermally stable at extremely high temperatures (at least ~1800 °C) [5]. Piezoelectric crystals of ZnO can transform mechanical energy into an electrical signal, or vice versa [18].

ZnO is generally classified as a colorless and low-toxicity material that does not cause skin or eye irritation; additionally, there is no evidence of carcinogenicity, genotoxicity, or toxicity in humans [19][20]. However, the dust can be hazardous by inhalation or ingestion [21], as it causes a condition known as zinc fever [5]. The current evidence shows that ZnO particles and nanoparticles do not penetrate skin cells and remain in the outer layer of undamaged skin (the stratum corneum) with low systemic toxicity [22]. ZnO is a material that can act as a catalyst in redox reactions that promote the photodegradation of pollutants [23].

Some aspects that motivate its use in binders and asphalt mixtures are as follows: (i) it is a material with high compatibility with asphalt and is stable at high temperatures [24][25][26]; (ii) it improves the UV aging resistance of asphalt [14]; (iii) adding nano-ZnO to asphalt mixtures reduces the acid component and increases the basic component of the binder, improving its adhesion with the aggregate [27]; (iv) photocatalytic infrastructures based on semiconducting nanoparticles such as ZnO can be promising solutions with which to improve urban air quality due to their ability to mineralize toxic organic compounds; (v) car exhaust components such as ozone (O_3), cyclooxygenase (COX), volatile organic compounds (VOCs), nitrogen oxides (NOX), carbon dioxide (CO_2), and nitric oxide (NO) emissions tend to decrease in presence of ZnO surfaces [28][29]; (vi) ZnO could remove acidic contaminants such as nitrate (NO_3) and sulfate (SO_4) from the water rainfall on the pavement surface [30].

2. ZnO as a Modifier of Binders and Asphalt Mixtures

To modify the asphalt binders, between 1 and 5 wt% ZnO was generally used. Regarding the modified binders, the most evaluated properties were conventional (penetration, softening point, etc.) and rheological. Few studies measured chemical properties. Regarding the modified asphalt mixtures, the resistance to aging and the properties at high service temperatures were mainly evaluated (few studies evaluated properties at low temperatures). ZnO tended to increase the resistance to aging, moisture damage, and rutting of binders and asphalt mixes.

The following is a summary of the information consulted chronologically.

Du et al. [16] evaluated the effect of nano-ZnO (15–25 nm) on the morphology and UV aging properties of two asphalt binders (AC 73 dmm and AC 92 dmm). Both AC were mixed with nano-ZnO (2 wt%) at 150 ± 5 °C at 5000 rpm for 60 min using a high shear mixer. Then, mixing was continued using a paddle agitator at 2000 rpm for 90 min. This same process was copied for the ACs without the nano-ZnO for comparison. The asphalt binders were aged in TFOT + UV. The TFOT residue was aged with UV for a period of 0 to 24 days in an oven with a 500 W UV lamp. The UV radiation intensity was 8 w/m^2. The penetration (25 °C), softening point, ductility (15 °C), RV (60 °C), and rheological properties were measured on the binders before and after aging using a DSR. Tests were also carried out using AFM and FTIR. The aging resistance of both binders improved, but the effect of nano-ZnO depended on the type of base binder.

Liu et al. [17] modified an AC 73 dmm with 3 wt% nano-ZnO (15–25 nm). The surface of the nanoparticles was previously modified with γ-methacryloyloxypropyltrimethoxysilane (MTS), 3-aminopropyltriethoxysilane (APTS), and γ-(2,3-epoxypropoxy) propyltrimethoxysilane (EPTMS). Mixing was performed at 150 ± 5 °C, 5000 rpm for 60 min, using a high shear mixer. Then, mixing was continued with a paddle stirrer at a speed of 2000 rpm for approximately 90 min. Researchers performed the HTS Test; aged the binders in TFOT + UV; and performed softening point, penetration, ductility (15 °C), RV (60, 135 °C), and FTIR tests. The resistance to UV aging improved with nano-ZnO, and the resistance changed depending on the type of modifier used on its surface. According to the researchers, surface modification of nano-ZnO was an effective method with which to improve the binder’s resistance to UV aging.

Zhang et al. [14] modified an AC 73 dmm with nano-ZnO (15–25 nm) and nano-ZnO modified on the surface with c-(2,3-epoxypropoxy) propyltrimethoxysilane. Mixing of AC with 2 wt% nano-ZnO (without and with modification) was performed at 150 ± 5 °C at 5000 rpm for 60 min using a high shear mixer. Then, mixing was continued using a paddle agitator at 2000 rpm for 90 min. The same process was performed on unmodified AC. The AC (unmodified and modified) was aged at TFOT + UV. The researchers performed softening point, penetration, ductility (5 °C), RV (60 and 135 °C), FTIR, AFM, and HTS tests. The compatibility between AC and nano-ZnO improved along with nano-ZnO surface modification. The UV aging resistance improved when AC was modified with nano-ZnO, and was higher when the latter was modified with c-(2,3-epoxypropoxy) propyltrimethoxysilane. On the other hand, in the same year, Zhang et al. [15] published a similar study using two other nanoparticles (nano-SiO_2 and nano-TiO_2). The conclusions in the latter study were the same as those in the former. However, Zhang et al. [15] concluded that, compared to the other two nanoparticles, the nano-ZnO-modified binder showed the best resistance to UV aging after surface modification.

Zhang et al. [31] modified an AC 75 dmm with nano-ZnO (15–25 nm) and OEVMT (sieve 300). The nano-ZnO surface had been previously modified with 3-aminopropyltriethoxysilane. The content of both nanomaterials was 3wt%; however, the individual content of nano-ZnO and EVMt was unclear. Mixing was performed at 150 ± 5 °C and 5000 rpm for 60 min using a high shear mixer. It was then mixed using a paddle stirrer at a speed of 2000 rpm for 90 min. The same process was also performed for the base binder. The modified binders were aged in PAV, and softening point, penetration, ductility (15 °C), RV (60, 135 °C), XRD, FTIR, and Ultraviolet visible (UV-Vis) diffuse reflectance spectra tests were performed. The nano-ZnO improved the resistance to UV aging.

Zhang et al. [32] modified an AC 63 dmm with 1 wt% OEVMT and 1, 2, 3, and 4 wt% nano-ZnO (average particle size 20 nm, which was previously surface-modified with c-(2,3-epoxypropoxy) propyltrimethoxysilane). Mixing was carried out at 150 °C at 4000 rpm for 60 min using a high shear mixer. Then, mixing was continued at a rotational speed of 2000 rpm at 150 °C for approximately 90 min. The same preparation process was also performed on the base binder. The binders were aged in TFOT, PAV, and UV. Penetration, softening point, RV (135 °C), and DSR tests were performed on the samples. OEVMT and nano-ZnO improved the resistance to thermal oxidation and aging. The recommended content of these materials was 1% OEVMT + 3% nano-ZnO. Nano-ZnO has a negative influence on the physical properties of the binder when its content exceeds 4 wt%.

Hamidi et al. [33] modified an AC 69 dmm with nano ZnO (2 and 4 wt%). The AC was mixed with nano ZnO for 4–5 min at 130–140 °C and 14,000 rpm. The researchers manufactured asphalt mixes designed by the Marshall procedure and measured the TSR and SFE on the samples. Nano ZnO decreased the acidity of the asphalt binder, improving the adhesion with an acid aggregate such as granite. It increased the SFE of adhesion between the aggregate and asphalt. This increased the moisture damage resistance of the asphalt mix. A similar study with the same conclusions can be found in Hamedi and Nejad [34]. However, in the latter study, the researchers evaluated the effect of adding another nanomaterial (nano calcium carbonate) and performed dynamic modulus tests.

Saltan et al. [35] modified an AC 62.2 dmm (PG 64–22) with ZnO (1, 3, and 5 wt%). Both materials were mixed at 4000 rpm and 160 °C for 2 h. The modified binder was aged in a RTFOT (rolling thin film oven test) and PAV. Its viscosity was measured, and its fatigue and rutting performances were evaluated via DSR and BBR tests. The researchers manufactured HMA and measured moisture susceptibility (ITS) by performing Lottman-modified tests. The best performance of the blends was obtained with the 5 wt% ZnO modification. At 1wt%, the mixture performed similarly to the control sample. The HMA experienced higher resistance against moisture for all modification percentages. ZnO modifications had lower fatigue performance, but the highest rutting and aging performances.

Xu et al. [36] modified an AC 69 dmm with nano-ZnO powder (average particle size 30 nm). The additional percentages were 1, 2, 3, 3, 4, and 5 wt%. The mixing was performed at 150 ± 5 °C at a rotation speed of 5000 rpm for 60 min and stirred with a paddle mixer at a rotation speed of 2000 rpm for 90 min. Penetration, softening point, ductility (5 °C), storage stability, DSR, and LAS tests were performed on the modified binder. Characterization tests were performed on TFOT and UV-aged samples. The nano-ZnO increased the stiffness of the binder and its resistance to UV aging. It was also able to significantly improve its rheological properties. The reasonable dosage of nano-ZnO was quantified at 3 wt%.

Fakhri and Shahryari [37] modified an AC 64 dmm with nano-ZnO (0.2, 0.4, and 0.6 wt%; particle diameter 20–60 nm) and nano-reduced graphene oxide (RGO). Mixing was carried out at 160 ± 5 °C and 4000 rpm for 50 min. The modified binders were tested for ductility (15 °C), Saybolt viscosity (135 °C), softening point, penetration, and specific gravity. The researchers manufactured SMA mixtures designed using the Marshall method and performed immersion Marshall, ITS, boiling water, static creep, pull-off adhesion, and SCB (25 °C) tests on them. Both nanomaterials increased the stiffness of the binder and improved the coating and adhesion with the aggregates. Additionally, both nanomaterials improved the performance of the mixes to moisture damage and rutting, and increased the fracture energy promoting resistance against fatigue crack propagation in dry and wet conditions. The best performance was obtained with 0.6 wt% addition of nano-ZnO.

He et al. [38] modified an AC 65 dmm with rodlike nano-ZnO (0.5, 1, 2, 2, 3, and 4 wt%). Mixing was performed at 150 ± 5 °C for 1 h at 5000 rpm, and the samples were aged in TFOT + UV. The researchers performed penetration tests as well as softening point, ductility (5 °C), DSR, FTIR, UV-visible-infrared-absorption, and SEM observations. The rodlike nano-ZnO increased the binder stiffness and resistance to UV aging. The best performance was reported for 3 wt% addition.

De Sousa Neto et al. [39] modified an AC 53 dmm with nano-ZnO (3, 5, 7 wt%). The mixing was performed at 150 ± 5 °C, 2000 rpm for 90 min. The researchers performed rheology tests (PG, MSCR, LAS, master curve), and the modified binders showed improved performance at high temperatures by increasing consistency; viscosity; stiffness; and resistance to aging, rutting, and fatigue. This was especially true at a nano-ZnO content of 7 wt%.

Du et al. [40] modified an AC 66.5 dmm with ZnO and vermiculite (EV) at ratios of 2, 3, 4, 5, 5, and 6 wt%. The ZnO/EV and binder were mixed at 150 °C for 1 h at 3600 rpm, and then mixing was continued using a mechanical stirrer at 2000 rpm for 1.5 h. The asphalts were aged in RTFOT, PAV, and UV. The researchers manufactured asphalt mix and subjected it to STOA. Penetration, softening point, ductility (15 °C), RV (135 °C), DSR, and BBR tests were performed on the modified asphalt binder. Marshall, low-temperature indirect tensile (−6, −12, −18 °C), freeze–thaw splitting, and indirect tensile fatigue (25 °C) tests were performed on the mix. ZnO/EV increased the binder stiffness and rutting resistance at high temperatures, but decreased its ductility and compromised its low-temperature performance. With 5% ZnO/EV, the modified asphalt mix achieved better high- and low-temperature performance, lower susceptibility to moisture, and higher fatigue resistance than the unmodified asphalt mix.

Fu et al. [41] modified an AC 85 dmm with nano-TiO_2, nano-ZnO (nano- TiO_2/ZnO = 2, 4, 6 and 8 wt%), and basalt fiber—BF (6 wt%). The nano- TiO_2 and nano-ZnO were mixed and stirred at a 1:3 ratio for 5 min at 160 ℃. Then, both materials were mixed with the asphalt binder for 10 min. Subsequently, shear was applied at 5000 rpm for approximately 50 min. Finally, BF was added and mixed at 2500 rpm for 40 min. The binders were aged at TFOT, PAV, and UV. DSR, ZSV, MSCR, LAS (25°C), BBR, FTIR tests and observations in SEM were performed on the modified binder. Nano-TiO_2, nano-ZnO, and BF increased rutting, intermediate temperature fatigue, and aging resistance. BF decreased low-temperature fatigue performance, but the addition of nano-TiO_2/ZnO may have helped to increase it. The optimum nano-TiO_2/ZnO dosage was 4wt%.

Kamboozia et al. [42] modified an AC 68 dmm with ZnO (2, 4, 6, and 8 wt%). Both materials were mixed at temperatures between 140 to 155 °C, under 2000 and 5000 rpm, for 60 to 70 min. To disperse the ZnO in the binder, Kamboozia et al.

[42] used kerosene as a solvent (it is not clear what the effect of this solvent had on the results). The binders were tested for their softening points, penetration, ductility, and RV (120, 135, 150 °C) to determine the optimum ZnO content (6 wt%). Porous mixtures were manufactured using a SGC and then designed using the Cantabro, Marshall, and ITS tests. Additional Wheel Track Test (45 °C) and SCB (−15, 0, 15 °C) tests were performed on the designed mix. The addition of ZnO increased the binder viscosity, adhesion, and cohesion of the mix. Additionally, it improved the rutting resistance of the porous mix. However, in the SCB test, it was found that the performance of the mixes with ZnO was not the best.

Guo and Zhang [43] modified an AC 64.3 dmm (PG 64-16) with different ZnO morphologies: agglomerate, sphere, rod, and flower. AC was modified with 2, 3, and 4 wt% of ZnO (particle average size was 2 μm). Initially, mixing was performed at 160 °C for 20 min (2000 rpm), and then mixing was conducted at 180 °C for 40 min (4000 rpm). Penetration, softening point, ductility (10 °C), RV (120 °C), DSR, and BBR tests were performed on the modified binders. The researchers manufactured asphalt mixtures designed by the Marshall method and measured the TSR, DS, flexural strength, and flexural tensile strain (three-point bending beam test) on them. ZnO increased the viscosity, stiffness, high-temperature PG, rutting, and aging resistance of the asphalt binder. However, the morphology of the ZnO particle had no effect on the softening point, ductility, penetration, anti-aging ability, or low-temperature ability of the asphalt binder. It also had no effect on the water stability of the asphalt mixture. ZnO particles could improve the DS and water stability of an asphalt mixture. When the morphology of ZnO is rod-like, the properties of asphalt binder and asphalt mixture are better.

Di et al. [44], using nano-ZnO (4 wt%) and Nano-TiO_2 (4 wt%), modified two ACs with two different wax contents, but the physical properties of the ACs are not clear. Researchers show that the rheological properties and wax content of both ACs are their main properties. The modification was performed at 155 °C and 5000 rpm for 30 min using a high shear mixer. LAS, MSCR, and NMR spectroscopy tests have been performed on the modified binders. Both additives improved the aging, rutting, and fatigue resistance of the binder, and nano-ZnO had a greater influence on the fatigue life of the asphalt compared to nano-TiO_2. The antioxidant mechanism of nano-TiO_2 is to inhibit the loss of aromatic carbon, while the oxidation of a nano-ZnO-modified binder causes sufficient aromatization to compensate for the influence of different degrees of aging.

Han et al. [45] initially modified an AC 70 dmm (PG 64-16) with a high content of linear elastomer SBS 791-H (6.5 wt%) by mixing them at 185 °C for 35 min at a speed of 5000 rpm. This SBS-binder was then modified with nano-ZnO, nano-TiO_2, and nano-SiO_2 to attempt to improve its anti-aging performance. The content of all anti-aging agents was 2 wt%. Rheology tests (DSR), FMI, and infrared spectroscopy tests were performed on the modified binders. The nanoparticles did not show significant effects on the rutting, the fatigue, or the aging resistance of the SBS-modified binder. This was mainly due to the weak physical interaction between the nanoparticles and the SBS. Furthermore, no obvious chemical reaction was observed.

Staub de Melo et al. [46] modified an AC 60 dmm (PG 58) with ZnO (3, 6, 9, 12, and 15 wt%). The modification was performed at a temperature of 150 ± 5 °C using a high shear mixture at a speed of 6000 rpm for 15 min. Staub de Melo et al. [46] used a DSR to measure the PG at high temperatures, then used aging index (AI) and non-recoverable creep compliance tests, as well as LAS (20 °C) tests. ZnO increased the stiffness, high-temperature PG, rutting, and aging resistance of the asphalt binder. The fatigue strength increased with ZnO at low strain amplitudes, but decreased at high strain amplitudes.

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