# Laboratory evaluation of antioxidant modified asphalt mixture

Muskan Verma
*Research Scholar, Department of Civil Engineering, Indian Institute of Technology Roorkee, Roorkee, INDIA- 24766, muskan_v@ce.iitr.ac.in*

Nikhil Saboo
*Associate Professor, Department of Civil Engineering, Indian Institute of Technology Roorkee, Roorkee, INDIA- 247667, nikhil.saboo@ce.iitr.ac.in*

ABSTRACT: Use of antioxidant is one of the effective alternatives to retard aging of binder, thereby enhancing the service life of asphalt pavements. Existing studies on the feasibility of antioxidants have demonstrated promising results at both macro and micro levels. Thus, to promote the wide application of antioxidant additives, it is imperative to evaluate the effect of modification at the mixture level. The present study focused on assessing the performance of a primary antioxidant (Irganox 1010) modified asphalt mixture. The critical characteristics, including stiffness, rutting, and fatigue were estimated by resilient modulus, wheel tracking, and indirect tensile cracking test (IDEAL-CT) tests. Laboratory findings revealed that addition of Irganox 1010 lowered the rate of long-term binder hardening of asphalt mixture. The modification enhanced the fatigue life of a control asphalt mixture by 10%, while ensuring satisfactory rutting resistance.

## 1 INTRODUCTION

The gradual changes occurring in the properties of asphalt binder and mixtures due to elevated temperature, UV radiation, or humidity is referred as aging. In general, the aging of binder starts at the mixing and paving stage due to volatilization of binder components, termed short-term aging (STA). This is followed by in-service oxidative aging of binder, known as long-term aging (LTA). With age, the binder becomes excessively stiff and brittle, increasing the cracking susceptibility of the asphalt mixture. This reduces the damage tolerance ability of binder leading to early distress formation in hot mix asphalt (HMA) pavement. Thus, necessitating the use of effective age retarding methods.

The incorporation of antioxidant (AO) additive in asphalt binder is one of the feasible alternatives to inhibit binder aging. Antioxidants are reducing agents that prevent the formation of oxidation propagating compounds (free radicals) in the autooxidation reaction. They function either as peroxide decomposers or free radical scavengers to abate the binder aging process. In general, phenolic, amine, phosphate, and sulphide groups are major anti-aging agents. Several commercially available AO additives widely used in polymer industries have been explored to abate the rate of binder aging (Verma and Saboo 2024). Numerous studies have evaluated the effectiveness of AO additives in enhancing the aging resistance of asphalt binders. Thus, to translate the laboratory evaluations to field, it is essential to assess the effect of AO on the mechanical characteristics of asphalt mixtures. The addition of additives such as olive pomace, lignin, and Irganox 1076 has been reported to improve the long-term cracking resistance of asphalt mixtures without compromising their rutting resistance (Verma and Saboo 2024). However, limited work has been conducted on the antioxidant modified asphalt mixtures. Therefore, it is crucial to have a comprehensive understanding of AO modification on the performance attributes of asphalt mixture. Thus, the present study aims to examine the changes in stiffness, rutting, fatigue, and aging characteristics of asphalt mixtures with and without AO modification.

## 2 MATERIALS

### 2.1 *Asphalt binder and aggregates*

In this study a viscosity graded (VG), VG-30 asphalt binder was selected as a base binder. The aggregates were obtained from a local quarry. The physical properties of procured binder and aggregates are presented in Table 1 and Table 2, respectively. The measured properties were observed to follow the desired specification (IS:73 2013; MoRTH 2013).

Table 1. Physical properties of VG 30

| Tests | Result | Limit |
|---|---|---|
| Penetration Test | 46 | 45 (min) |
| Softening point test ($^0$C) | 51 | 47 (min) |
| Absolute viscosity (poises) at 60 $^0$C | 2899 | 2400 - 3600 |
| Absolute viscosity (poises) RTFO aged bitumen | 10877 | - |
| Viscosity Ratio at 60 $^0$C | 3.75 | 4 (max) |
| Ductility at 25 $^0$C, cm RTFO aged sample | > 100 | 40 (min) |

* min: minimum; max: maximum

Table 2. Physical properties of mineral aggregates

| Properties | Value | Limit |
|---|---|---|
| Aggregate Impact Value, % | 18 | 27 |
| Los Angeles Abrasion Value, % | 30 | 35 |
| Combined flakiness and elongation index, % | 22 | 35 |
| Water Absorption | 0.3 | < 2 % |
| Specific gravity (CA) | 2.647 | - |
| Specific gravity (FA) | 2.556 | - |
| Specific gravity (Filler) | 2.541 | - |

## 2.2 *Antioxidant additive*

A sterically hindered phenolic compound, Irganox 1010 (pentaerythritol tetrakis 3-(3,5- di-tert-butyl 4-hydroxyphenyl) propionate) was used for binder modification. It is a type of primary antioxidant that mitigates the chain reaction of aging by the free radical scavenging mechanism. It is widely applied to prevent degradation of polymer and rubber made materials. Table 3 presents the general properties of the Irganox 1010 additive used in present work.

Table 3. Properties of Irganox (1010) additive

| **Melting point** | **110 – 125 °C** |
|---|---|
| Appearance | White powder form |
| Purity | 99 % |
| Specific gravity (g/mm, 20 $^0$C) | 1.116 |

# 3 SAMPLE PREPARATION

At first, the base binder was modified with 0.4% (by weight of binder) of Iragnox (1010). This dosage was considered based on the literature review. The existing studies have suggested that the dosage >1% softens the binder. Varying the dosage between 0.4% to 0.6% was observed to achieve optimum binder performance results (Apeagyei 2011). Therefore, the modified binder was prepared by blending the additive in the preheated binder at 125 °C for 30 minutes using a high-shear mixer at 1000 rpm speed.

Further, the asphalt mixtures were prepared with desired gradation of bituminous concrete (BC-1), as shown in Figure 1. BC-1 grading is employed as a wearing course in Indian highways. It is the most susceptible layer to get damaged due to aging. The Marshall mix design was carried out as per MS-2 (Asphalt Institute 2009) to determine the optimum binder content (OBC) of the mixtures. The OBC corresponding to 4% target air voids was found to be 5.21% (by weight of the mix). To assess the effect of antioxidant modification, both the modified (I(0.4)) and control mixtures (VG 30) were prepared at the same OBC. Subsequently, the samples were subjected to laboratory aging asper AASHTO R30 (AASHTO 2015). To simulate the STA, the prepared loose asphalt mixtures were aged for four hours at 135°C, followed by five-day conditioning of compacted samples at 85°C for LTA.

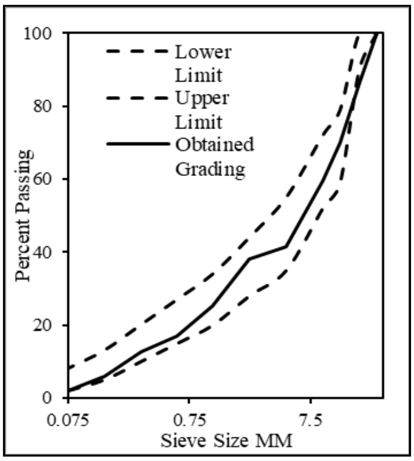


Figure 1. Selected aggregate gradation BC-1

# 4 METHODOLOGY

Laboratory experiments were conducted to estimate the effect of AO modification on the performance of HMA mixes. To ensure repeatability at least two replicates of each mixture were tested. Figure 2 shows the comprehensive experimental program followed to address the objective of the study.

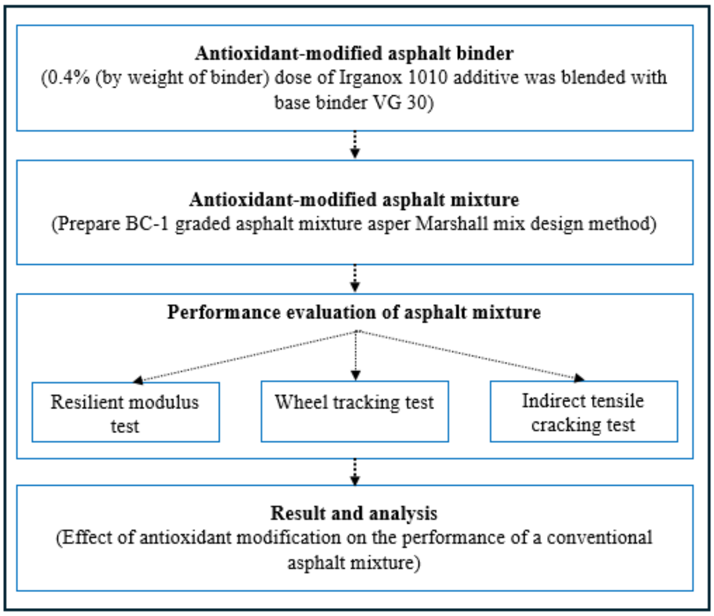


Figure 2. Methodology flow chart

## 4.1 *Resilient modulus*

Resilient modulus ($M_r$) is a parameter used to determine the stiffness characteristics of an asphalt mixture. To evaluate the effect of aging, the asphalt mixtures were tested at each aging condition (STA and LTA). The repeated-load indirect tension test as per ASTM D4123 (ASTM 1995) was employed to evaluate Mr at 35°C. The cylindrical specimen was subjected to a haversine compressive loading for 0.1 seconds followed by a rest period of 0.9 seconds. The resulting recoverable horizontal deformation at the diametrical plane of the specimen was measured using two linear variable displacement transducers (LVDT's).

## 4.2 *Rutting resistance*

Wheel tracking test was adopted to evaluate the rutting susceptibility of the asphalt mixtures (as per BSI EN 12697-35 (BS 2020). The test setup consists of a steel wheel and a modified Marshall sample of 150 mm diameter and 50 mm height, prepared at 7 ± 0.5% air void. A vertical load of 700 ± 10N was applied by a lever arm mechanism. The accumulated deformations at five different locations were recorded up to the limiting criteria of 12 mm total rut depth or 20,000 load passes are completed. The performance was measured for STA conditioned asphalt mixture at 60 °C, to simulate the maximum in-service pavement temperature.

## 4.3 *Fatigue resistance*

The indirect tensile cracking test (IDEAL-CT) is a simple test used to estimate the cracking resistance of asphalt mixture. The test was performed on LTA conditioned samples at an intermediate temperature of 25°C asper ASTM D8225 (ASTM 2019). The cylindrical Marshall specimen was prepared at 7% air void and long-term aged to surrogate the in-field pavement conditions. A vertical load was applied on the diametrical axis at a constant deformation rate of 50 ± 2 mm/min and a load versus displacement curve was plotted. The $l_{75}/m_{75}$ (mm/kN) ratio was used to indicate the flexibility of asphalt mixture. It is found sensitive to field aging and thus considered a desirable parameter to simulate the field cracking performance of asphalt mixture (Leavitt et al. 2024). In general, with increasing aging severity the flexibility parameter, $l_{75}/m_{75}$ ratio decreases. The $l_{75}/m_{75}$ ($mm^2$/kN) ratio is calculated from the post-peak analysis of the load-displacement curve. Wherein, $l_{75}$ presents the displacement corresponding to 75% of peak load (mm) and $m_{75}$ is the absolute value of the slope (N/m)

## 5.1 *Resilient modulus*

The resilient modulus result describes the stiffness of asphalt mixtures under repeated load application. The $M_r$ value of asphalt mixture increased with aging severity, as shown in Figure 3. At STA level, a slight increase of 2% in the stiffness was observed for I (0.4) mixes. Whereas, for LTA conditioned samples, the stiffness of modified mixture decreased by 15%. The rate of stiffness increment from STA to LTA condition was 8% for AO modified mix and 30 % for the base binder, indicating improvement in aging resistance after modification. This could be attributed to the peptizing action of Irganox 1010 additive which inhibits the gelatinization of aged asphalt binder. It can therefore be concluded that the incorporation of antioxidant additive lowered the rate of aging, without affecting the stiffness at the initial STA level.

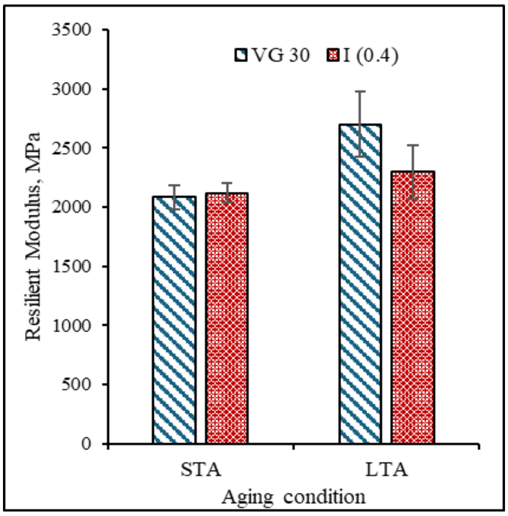


Figure 3. Resilient modulus results

## 5.2 *Rutting*

Rutting is a high temperature distress primarily observed in the early life of pavement. Therefore, it is imperative to evaluate the effect of AO modification on the rutting performance of asphalt mixture. Figure 4 shows the variation of rut depth with number of load passes. At the end of 20000 passes, the average total accumulated rut depth was recorded as 5.33 mm and 4.87 mm for VG 30 and I (0.4) asphalt mixtures, respectively. Thus, implying that rutting performance was not compromised due to antioxidant modification.

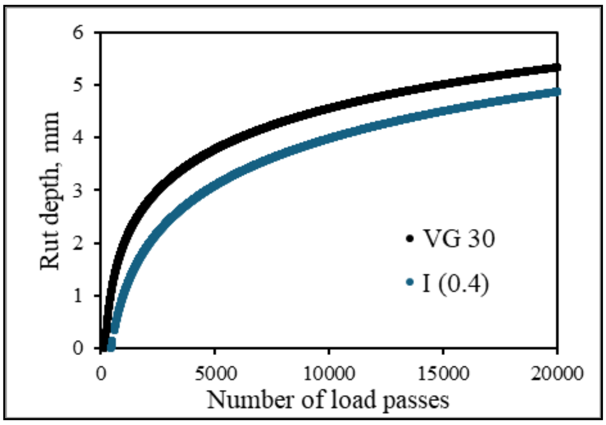


Figure 4. Rutting test results

## 5.3 *Fatigue*

Antioxidant additives inhibit aging of asphalt binder and are thereby expected to improve the cracking resistance of asphalt mixtures. Figure 5 presents the load-displacement curve of both VG 30 and I (0.4) asphalt mixes. As can be seen, compared to the control mix, a 10% increase in $l_{75}/m_{75}$ parameter was observed after modification. This signifies that antioxidant modification elevates the flexibility and the fatigue performance of asphalt mixtures.

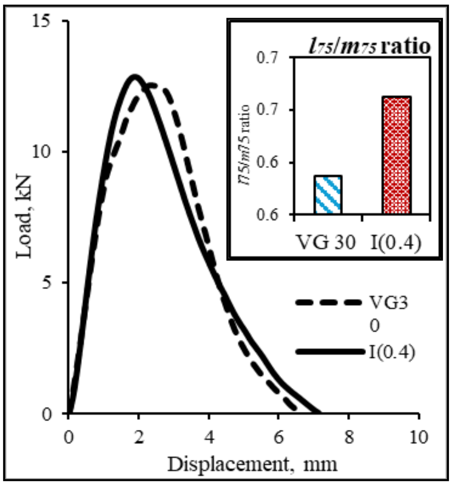


Figure 5. IDEAL-CT results

# 6 CONCLUSION

In summary, the results revealed that the incorporation of Irganox 1010 additive improved the long-term performance of asphalt mixes without negatively affecting its rutting performance. Based on the results, the following conclusions were drawn:

- In terms of stiffness, the addition of Irganox 1010 decreased the long-term stiffness of the control asphalt mixture by 15%.
- The stiffness increment due to aging was found to be 30% and 8% before and after AO modification, respectively. This implies lower binder hardening and better aging resistance.
- The rutting resistance of AO modified mixture was found comparable to the control mix.
- Compared to control mix, the $l_{75}/m_{75}$ ratio was observed to increase by 10% after modification, exhibiting better fatigue performance.

# 7 REFERENCE


AASHTO. 2015. “AASHTO R30: Mix conditioning of hot mix asphalt (HMA), Washington, DC.” *AASHTO*.

Apeagyei, A. K. 2011. “Laboratory evaluation of antioxidants for asphalt binders.” *Constr. Build. Mater.*, 25 (1): 47–53. Elsevier Ltd. https://doi.org/10.1016/j.conbuildmat.2010.06.058.

Asphalt Institute. 2009. “MS-2 Asphalt Mix Design Methods.” *ASTM Int.*, (111): 1–197.

ASTM. 1995. “ASTM D4123-20: Standard Test Method for Indirect Tension Test for Resilient Modulus of Bituminous Mixtures.” *ASTM Int.*, 82: 2–5.

ASTM. 2019. “Astm D8225, Standard Test Method for Determination of Cracking Tolerance Index of Asphalt Mixture Using the Indirect Tensile Cracking Test at Intermediate Temperatures.” *ASTM Int.*, 1–17.

BS. 2020. “EN 12967-22: Bituminous mixtures. Test methods. Part 22, Wheel tracking.” *BSI*, 32.

IS:73. 2013. “Paving bitumen - specification. New Delhi, India,Bur.Indian Stand.” (1–4).

Leavitt, A., A. Epps Martin, and E. Arámbula. 2024. “Model for Evaluating Cracking Performance of Asphalt Pavements with Field Aging Based on IDEAL-CT Parameters.” *Transp. Res. Rec.* SAGE Publications Ltd. https://doi.org/https://doi.org//10.1177/03611981241231806.

MoRTH. 2013. *Specifications for Road Bridge Works 5th Revision.*

Verma, M., and N. Saboo. 2024. “Use of antioxidants to retard aging of bitumen: A review.” *Environ. Sci. Pollut. Res. 2024 3136*, 31 (36): 48839–48863. Springer. https://doi.org/https://doi.org//10.1007/S11356-024-34431-2.