# Effect of Graphene-Based Nanocomposites on the Performance Characteristics of Bituminous Mixes

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**Abstract**

Warm mix asphalt (WMA) reduces production temperature and emissions compared with hot mix asphalt (HMA), but concerns remain about its moisture susceptibility and durability. This study produces WMA without chemical additives by a two-phase mixing process (TPMP), in which aggregates are pre-coated with a medium-setting bitumen emulsion and then mixed with VG-30 bitumen at reduced temperature. The binder is then modified with graphene oxide (GO) and reduced graphene oxide (RGO). From HMA trials, 8% GGBFS was selected as the best filler, giving a Marshall stability of 14.04 kN. Among WMA mixes made at 110, 120 and 130 °C with bitumen-to-emulsion ratios (BTER) of 100:0 to 50:50, the mix at 120 °C and 80:20 performed best. It gave a stability of 12.35 kN, a TSR of 88%, a retained Marshall stability of 76.54% and a rut depth of 4.12 mm. Adding GO and RGO at 0.1–0.5% by weight of bitumen raised the softening point and reduced penetration and ductility, and 0.3% was optimum for both. At 0.3%, the GO- and RGO-modified WMA reached stabilities of 16.28 and 16.13 kN, about 31% above the unmodified WMA. The GO-modified WMA gave an ITS of 680 kPa and a TSR of 88.3%, a rut depth of 3.91 mm at 50 °C (close to 3.48 mm for HMA) and a ravelling loss of 11.8%, 35% lower than HMA. Graphene-modified WMA prepared by TPMP thus offers HMA-comparable performance at a lower production temperature.

**Keywords:** Warm mix asphalt; Graphene oxide; Reduced graphene oxide; Bitumen emulsion; Two-phase mixing process; GGBFS filler; Rutting; Moisture susceptibility

## 1. Introduction

Bituminous mixes are the most widely used material for flexible pavements, but rising traffic, heavier axle loads, temperature extremes and moisture cause rutting, fatigue and thermal cracking, ravelling and stripping. Conventional hot mix asphalt (HMA) is produced at about 150–165 °C, which consumes fuel, emits greenhouse gases and causes short-term binder ageing. Warm mix asphalt (WMA) is produced 30–40 °C lower, through foaming, organic additives, chemical additives or emulsion-based processes. Its performance, especially its moisture resistance and rutting resistance, is often below that of HMA.

Graphene-based nanomaterials such as graphene oxide (GO) and reduced graphene oxide (RGO) have a very high surface area and stiffness and can modify the stiffness, temperature susceptibility, adhesion and ageing resistance of bitumen. GO carries oxygen-containing groups that can interact with bitumen components, while RGO restores much of the $sp^2$ carbon structure of graphene. The objectives of this study are to (i) select the best filler and determine the optimum binder content for HMA, (ii) develop additive-free WMA by a two-phase mixing process and identify the best mixing temperature and BTER, (iii) evaluate GO- and RGO-modified bitumen at 0.1–0.5%, and (iv) compare the performance of GO- and RGO-modified WMA with HMA and unmodified WMA.

## 2. Literature Review

Research on graphene in asphalt has progressed from binder modification to multi-scale characterisation and mixture-level performance. Behera et al. developed predictive models for graphene-modified WMA that integrate rheology and stress sensitivity [1]. Zhao et al. related the micro-mechanism of GO-framework-modified asphalt to road performance through grey correlation analysis [2]. Li et al. used FTIR to characterise the ageing of GO-modified binder [3], and Guo et al. combined GO with polyurethane as a composite modifier [5]. Uniform dispersion is critical to performance. Zhu and Zhang proposed an aerosol-based method to disperse graphene in asphalt emulsion [4], and Wang et al. examined the dispersion, compatibility and rheology of graphene-modified binders [12].

At the mixture level, Hoang et al. evaluated GO-modified asphalt concrete through a multi-test approach [9]. Polaczyk et al. reported laboratory evidence of improved pavement performance with graphene-modified asphalt [11], and Maltinti et al. presented a field case study of a graphene-modified thin layer [6]. Hamedi et al. showed that nano-GO mitigated the adverse effect of runoff pH on the moisture susceptibility of asphalt concrete [8]. Sarkar and co-workers evaluated warm-mix additives and graphene nanoplatelets for cracking resistance, moisture susceptibility and cost [7], [10]. However, few studies combine additive-free, emulsion-based WMA with GO and RGO binder modification, which this study addresses.

## 3. Materials and Methodology

### *3.1 Materials*

Crushed siliceous stone aggregates were used in a dense bituminous macadam (DBM) Grade-II gradation with a nominal maximum size of 19 mm (Fig. 1); their properties are given in Table 1. Granite stone dust, OPC and GGBFS (specific gravities 2.67, 3.0 and 2.9) were evaluated as fillers at 4, 6 and 8% by weight of aggregate. VG-30 bitumen conforming to IS 73 (Table 2) and a cationic medium-setting bitumen emulsion (Table 3) were used. GO (one to three layers) and RGO (three to six layers) were obtained as powders; their properties are given in Table 4 and shown in Fig. 2.

**Table 1.** Physical properties of aggregates

| Test | Result | Limit | Code |
|---|---|---|---|
| Impact value (%) | 18.1 | 27 max. | IS 2386 (P-4) / ASTM C131 |
| Crushing value (%) | 19.1 | 30 max. | IS 2386 (P-4) / ASTM D5821 |
| Los Angeles abrasion (%) | 17.7 | 35 max. | IS 2386 (P-4) / ASTM C131 |
| Combined FI and EI (%) | 22.9 | 35 max. | IS 2386 (P-1) / ASTM D4791 |
| Specific gravity | 2.74 | 2.5–3.0 | IS 2386 (P-3) / ASTM C127 |
| Water absorption (%) | 0.1 | 2 max. | IS 2386 (P-3) / ASTM C127 |
| Soundness (%) | 2.81 | 12 max. | ASTM C88 |
| Stripping value (%) | 2 | < 5 | IS 6241 / ASTM D3625 |

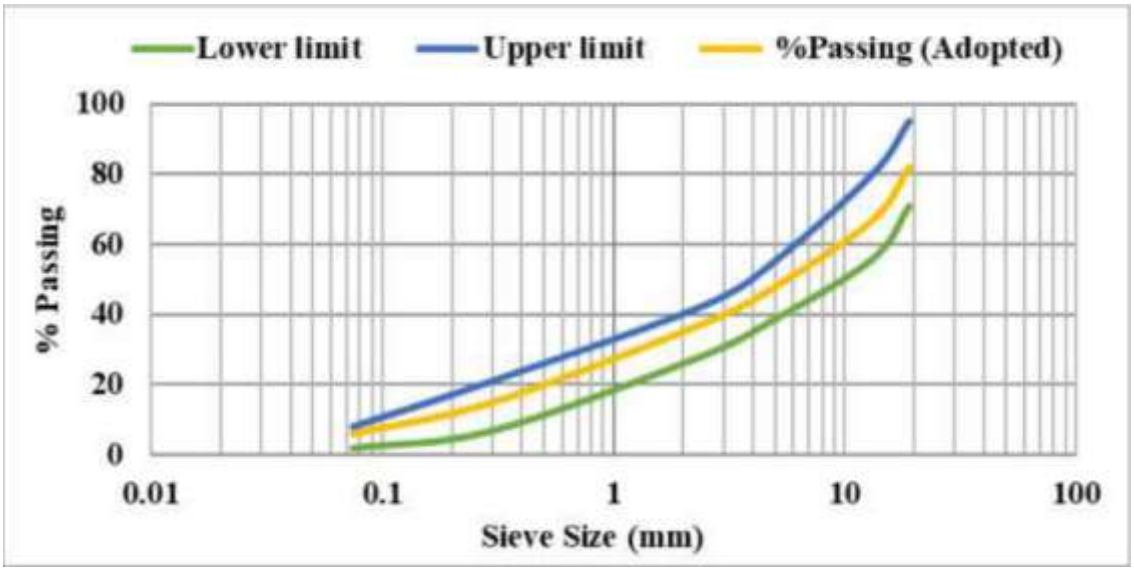



**Fig. 1.** Aggregate gradation for the DBM Grade-II mix

**Table 2.** Physical properties of VG-30 bitumen

| Test | Result | Limit | Code |
|---|---|---|---|
| Penetration at 25 °C (0.1 mm) | 63 | 50–70 | IS 1203 / ASTM D5 |
| Softening point (°C) | 50 | min. 47 | IS 1205 / ASTM D36 |
| Ductility (cm) | 65 | min. 47 | IS 1208 / ASTM D113 |
| Specific gravity | 0.99 | 0.97–1.02 | IS 1202 / ASTM D70 |

**Table 3.** Physical properties of the bitumen emulsion (IS 8887 / ASTM D2397)

| Test | Result | Limit |
|---|---|---|
| Saybolt Furol viscosity at 50 °C (s) | 130 | 50–300 |
| Residue by evaporation (%) | 65 | min. 65 |
| Residue penetration at 25 °C (0.1 mm) | 100 | 60–150 |
| Residue ductility at 27 °C (cm) | 100 | > 50 |

**Table 4.** Physical characteristics of GO and RGO (manufacturer data)

| Property | GO | RGO |
|---|---|---|
| Form | Powder | Powder |
| Colour | Black | Greyish black |
| Bulk density (g/cc) | 0.48 | 0.49 |
| Number of layers | 1 to 3 | 3 to 6 |
| Thickness | 2–4 nm | ~0.8–2 nm |
| Length | 5–10 µm | 6–10 µm |
| Carbon purity | ~99% | ~98% |
| Surface area (BET) | > 200 $m^2/g$ | ~130 $m^2/g$ |

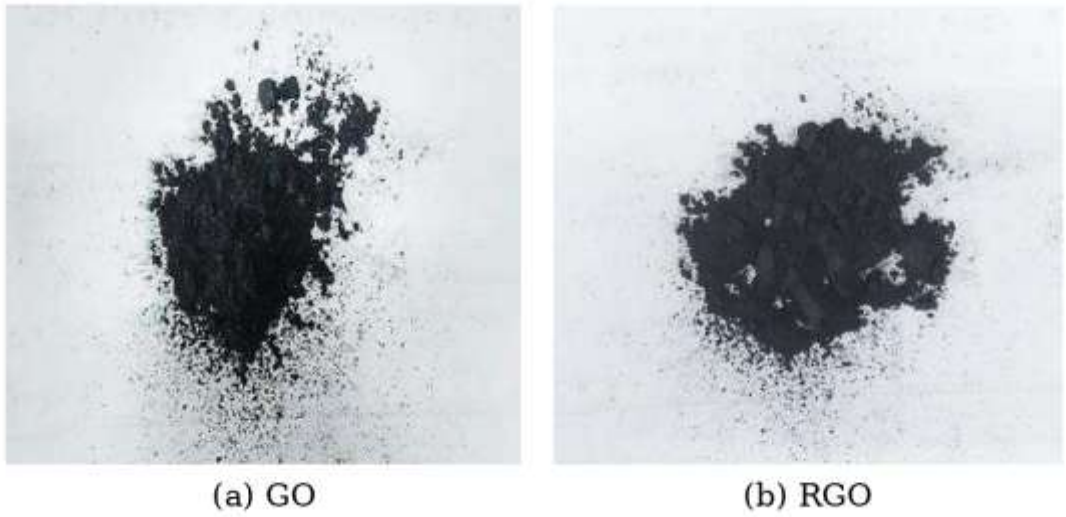



**Fig. 2.** Physical appearance of (a) GO and (b) RGO

### *3.2 Methodology*

**HMA.** Marshall specimens (75 blows per face) were prepared with 4.0–6.0% VG-30 bitumen heated to 165 °C, first for a control mix and then with each filler, to determine the optimum binder content (OBC) and the best filler type and content as per IRC:111-2009.

**WMA by TPMP.** Aggregates and 8% GGBFS were first coated with the medium-setting emulsion, supplying 10–50% of the total bitumen; because the emulsion has 65% residue, 10% of the bitumen required 15.4% emulsion. The remaining bitumen, as VG-30 heated to 130–140 °C, was then added to the pre-coated aggregates preheated for 30 minutes. The mix was stirred for 15–20 minutes at mixing temperatures of 110, 120 and 130 °C. The BTER was varied from 100:0 to 50:50.

**GO/RGO-modified bitumen.** GO and RGO were added at 0.1–0.5% by weight of 200 g of bitumen at 165 °C, in small portions to avoid agglomeration, and stirred with a mechanical stirrer at 1500 rpm for 20–40 minutes (Fig. 3). Modified binders were tested for softening point, penetration and ductility before and after RTFO and PAV ageing.

**Performance tests.** Mixes were evaluated for Marshall properties, indirect tensile strength (ITS) at 25 °C, tensile strength ratio (TSR), retained Marshall stability (RMS), wheel-tracking rut depth at 50 and 60 °C, Cantabro ravelling loss at 25 °C in the Los Angeles machine without abrasive charge (ASTM D7064), and semi-circular bending (SCB) fracture resistance at notch depths of 15, 20 and 25 mm.




**Fig. 3.** Preparation procedure of GO- and RGO-modified bitumen

## 4. Results and Discussion

### *4.1 Selection of Filler for HMA*

The Marshall parameters of HMA with cement and GGBFS fillers are given in Table 5. Stability increased with filler content for both fillers, and GGBFS gave consistently higher stability than cement. With 8% GGBFS, the stability reached 14.04 kN, 27.28% and 13.39% higher than HMA with granite stone dust and cement fillers. Its OBC was 5.01%, and its air voids, VFB and VMA were within limits. GGBFS at 8% was therefore selected as the filler for all subsequent mixes.

**Table 5.** Marshall parameters of HMA with cement and GGBFS filler

| Filler type and amount | Max. stability (kN) | Flow (mm) | Air voids (%) | Bulk density (g/cc) | VFB (%) | VMA (%) |
|---|---|---|---|---|---|---|
| Cement @ 4% | 10.89 | 2.99 | 3.025 | 2.36 | 77.55 | 14.46 |
| Cement @ 6% | 11.35 | 3.08 | 3.11 | 2.36 | 77.92 | 14.59 |
| Cement @ 8% | 12.16 | 3.11 | 3.17 | 2.37 | 78.10 | 14.47 |
| GGBFS @ 4% | 13.21 | 2.74 | 3.01 | 2.37 | 77.54 | 14.18 |
| GGBFS @ 6% | 13.56 | 2.79 | 3.02 | 2.38 | 77.95 | 14.20 |
| GGBFS @ 8% | 14.04 | 2.98 | 3.17 | 2.39 | 78.10 | 14.47 |

### *4.2 WMA by Two-Phase Mixing*

The best BTER was 70:30 at 110 °C, 80:20 at 120 °C and 90:10 at 130 °C (Table 6). At lower temperatures more emulsion was needed for coating, while at higher temperatures less emulsion and more hot bitumen gave the best result. The mix at 120 °C with 80:20 BTER was the best overall, with the highest stability (12.35 kN) and unit weight (24.35 kN/m³), air voids of 3.01% and an OBC of 5.08%. Its moisture resistance was also the best of the WMA mixes (Table 7). Its ITS was 635 kPa, its TSR 88% (close to 90% for HMA) and its RMS 76.54%. At 130 °C the RMS fell below the 75% minimum.

**Table 6.** Marshall parameters and OBC of WMA at different mixing temperatures (optimum BTER)

| Mixing temperature and BTER | Unit wt. (kN/m³) | Stability (kN) | Air voids (%) | Flow (mm) | OBC (%) |
|---|---|---|---|---|---|
| WMA @ 110 °C (70B:30E) | 24.10 | 11.30 | 4.31 | 2.45 | 4.98 |
| WMA @ 120 °C (80B:20E) | 24.35 | 12.35 | 3.01 | 2.20 | 5.08 |
| WMA @ 130 °C (90B:10E) | 24.12 | 11.10 | 4.01 | 2.60 | 5.35 |

**Table 7.** ITS, TSR and retained Marshall stability of HMA and WMA

| Mix | ITS (kPa) | TSR (%) | Stability after 30 min at 60 °C (kN) | Stability after 24 h at 60 °C (kN) | RMS (%) |
|---|---|---|---|---|---|
| HMA @ GGBFS | 808 | 90 | 14.04 | 11 | 78.34 |
| WMA @ 110 °C | 515 | 85 | 11.24 | 8.5 | 75.62 |
| WMA @ 120 °C | 635 | 88 | 12.15 | 9.3 | 76.54 |
| WMA @ 130 °C | 425 | 80 | 11 | 7.6 | 69.09 |
| MoRTH limit | — | min. 80 | — | — | min. 75 |

In wheel-tracking tests at 50 °C (Fig. 4), HMA with 8% GGBFS had a rut depth of 3.29 mm. WMA at 120 °C gave 5.26, 4.81, 4.12, 10.72, 15.23 and 16.72 mm for BTER of 100:0, 90:10, 80:20, 70:30, 60:40 and 50:50. The 80:20 mix was therefore closest to HMA, and higher emulsion fractions left more residual water and a softer mix. The 120 °C mix also had the lowest ravelling loss among the WMA mixes (Fig. 5). In SCB tests the peak load was highest at the 15 mm notch, and WMA carried lower peak loads than HMA at all notch depths.

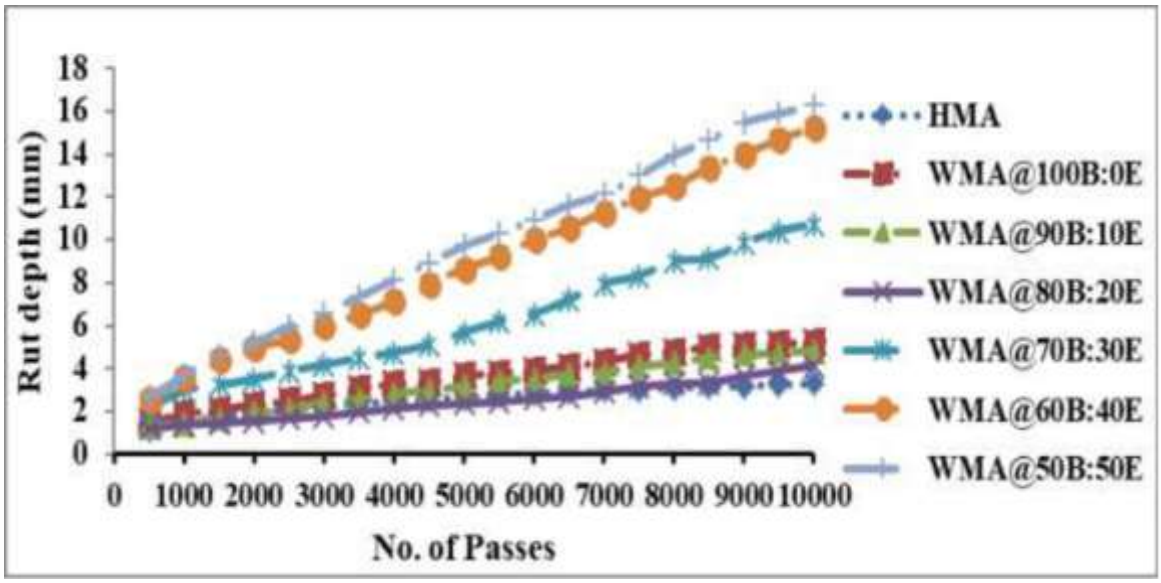



**Fig. 4.** Wheel-tracking rut depth versus number of passes for HMA and WMA at 120 °C with varying BTER

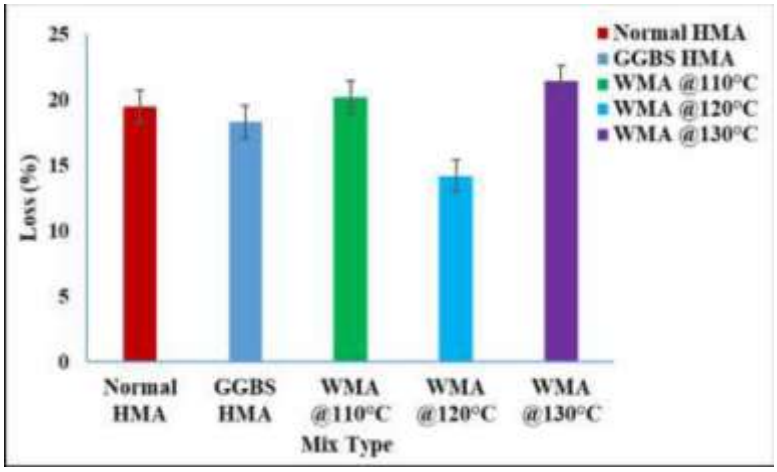



**Fig. 5.** Cantabro loss of HMA and WMA samples

### *4.3 Properties of GO- and RGO-Modified Bitumen*

Adding GO or RGO raised the softening point and reduced penetration and ductility in both the unaged and PAV-aged states (Table 8). At 0.3%, the softening point rose from 50 °C to 55 °C (GO) and 54 °C (RGO), and penetration fell from 61 to 55 and 53 (0.1 mm). The nanosheets stiffen the binder and reduce its temperature susceptibility, which benefits rutting resistance. The loss of ductility, especially after PAV ageing, indicates that dosages beyond about 0.3% risk making the binder too stiff. The physical properties alone did not identify an optimum, so it was selected from the mixture performance.

**Table 8.** Physical properties of unaged and PAV-aged GO- and RGO-modified bitumen

| Modifier and dosage (%) | Softening point unaged (°C) | Softening point PAV-aged (°C) | Penetration unaged (0.1 mm) | Penetration PAV-aged (0.1 mm) | Ductility unaged (cm) | Ductility PAV-aged (cm) |
|---|---|---|---|---|---|---|
| GO 0 | 50 | 52 | 61 | 55 | 78.5 | 40 |
| GO 0.1 | 51 | 54 | 58 | 49 | 63 | 35 |
| GO 0.2 | 52.5 | 56 | 56 | 46 | 55 | 25 |
| GO 0.3 | 55 | 58 | 55 | 44 | 50 | 20 |
| GO 0.4 | 56 | 59 | 53 | 41 | 47 | 16 |
| GO 0.5 | 57 | 61 | 51 | 39 | 41 | 12 |
| RGO 0 | 50 | 52 | 61 | 55 | 78.5 | 40 |
| RGO 0.1 | 51.5 | 56 | 59 | 47 | 61 | 38 |
| RGO 0.2 | 53 | 57 | 54 | 45 | 54 | 29 |
| RGO 0.3 | 54 | 59 | 53 | 42 | 48 | 24 |
| RGO 0.4 | 56 | 61 | 52 | 40 | 45 | 21 |
| RGO 0.5 | 58 | 62 | 50 | 38 | 39 | 16 |

### *4.4 Performance of GO- and RGO-Modified WMA*

Modified WMA was prepared at 120 °C with 80:20 BTER and 8% GGBFS. Stability peaked at 0.3% for both modifiers (Table 9), at 16.28 kN for GO and 16.13 kN for RGO. These are about 32% and 31% above the unmodified WMA (12.35 kN) and also above HMA (14.04 kN), with the highest Marshall quotients (4.99 and 4.78 kN/mm). Beyond 0.3%, stability fell and flow rose, especially with RGO, indicating agglomeration of the nanosheets. 0.3% was therefore taken as the optimum for both.

**Table 9.** Marshall parameters of GO- and RGO-modified WMA

| Dosage (% of bitumen) | GO stability (kN) | GO flow (mm) | GO MQ (kN/mm) | RGO stability (kN) | RGO flow (mm) | RGO MQ (kN/mm) |
|---|---|---|---|---|---|---|
| 0.1 | 14.64 | 3.42 | 4.28 | 14.40 | 4.82 | 2.98 |
| 0.2 | 15.61 | 3.29 | 4.74 | 14.92 | 4.12 | 3.62 |
| 0.3 | 16.28 | 3.26 | 4.99 | 16.13 | 3.37 | 4.78 |
| 0.4 | 15.33 | 3.67 | 4.17 | 13.01 | 5.25 | 2.47 |
| 0.5 | 14.57 | 4.50 | 3.23 | 12.31 | 5.62 | 2.19 |

The performance of the optimum mixes is compared in Table 10. GO modification raised the ITS of WMA from 635 to 680 kPa and the TSR to 88.3%, and RGO raised the RMS to 79.21%. All values met the MoRTH limits. Rutting resistance improved markedly (Fig. 6). At 50 °C the GO-modified WMA had a rut depth of 3.91 mm, against 5.52 mm for unmodified WMA and 3.48 mm for HMA. At 60 °C the GO-modified mix had 11.16 mm against 14.79 mm for unmodified WMA. The stiffer graphene-modified binder resists shear flow at high temperature. Ravelling loss fell from 18.3% for HMA and 14.2% for WMA to 11.8% and 12.65% for GO- and RGO-modified WMA (Fig. 7). The GO-modified mix lost 35% less than HMA, reflecting better cohesion and binder–aggregate adhesion. In SCB tests (Fig. 8), both modified mixes carried higher peak loads than unmodified WMA at every notch depth, approaching HMA. GO generally performed slightly better than RGO, probably because its oxygen-containing groups interact more strongly with bitumen and aggregate.

**Table 10.** Performance of HMA, WMA and 0.3% GO- and RGO-modified WMA

| Property | HMA | WMA | GOMB-WMA | RGOMB-WMA |
|---|---|---|---|---|
| ITS (kPa) | 808 | 635 | 680.33 | 657 |
| TSR (%) | 90 | 88 | 88.33 | 87.47 |
| Retained Marshall stability (%) | 80.34 | 76.54 | 77.12 | 79.21 |
| Rut depth at 50 °C (mm) | 3.48 | 5.52 | 3.91 | 5.22 |
| Rut depth at 60 °C (mm) | 8.33 | 14.79 | 11.16 | 13.12 |
| Ravelling (Cantabro) loss (%) | 18.3 | 14.2 | 11.8 | 12.65 |

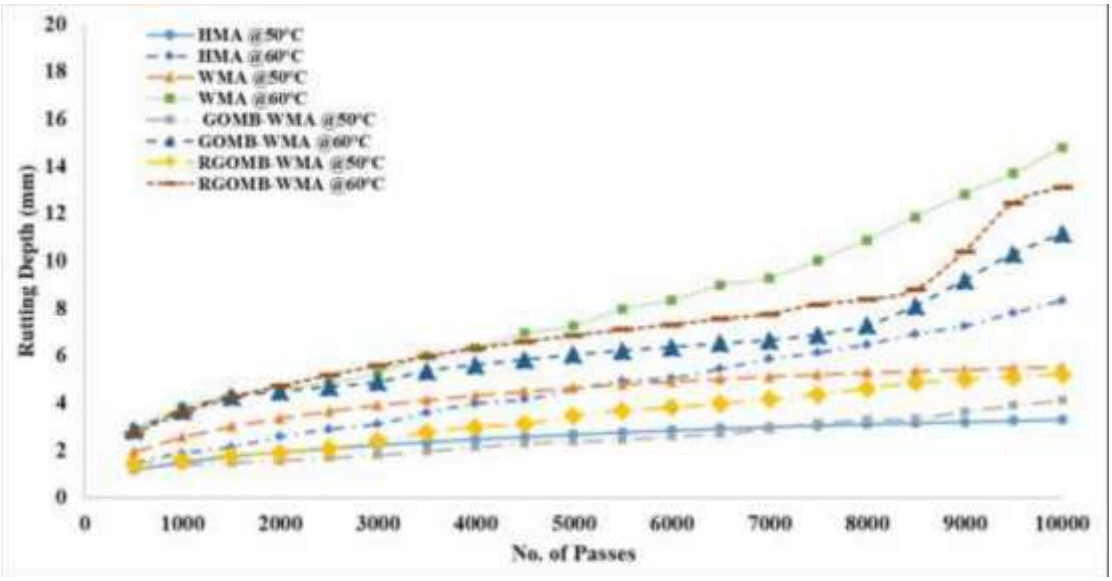



**Fig. 6.** Wheel-tracking rut depth versus load passes for HMA, WMA and GO- and RGO-modified WMA at 50 °C and 60 °C

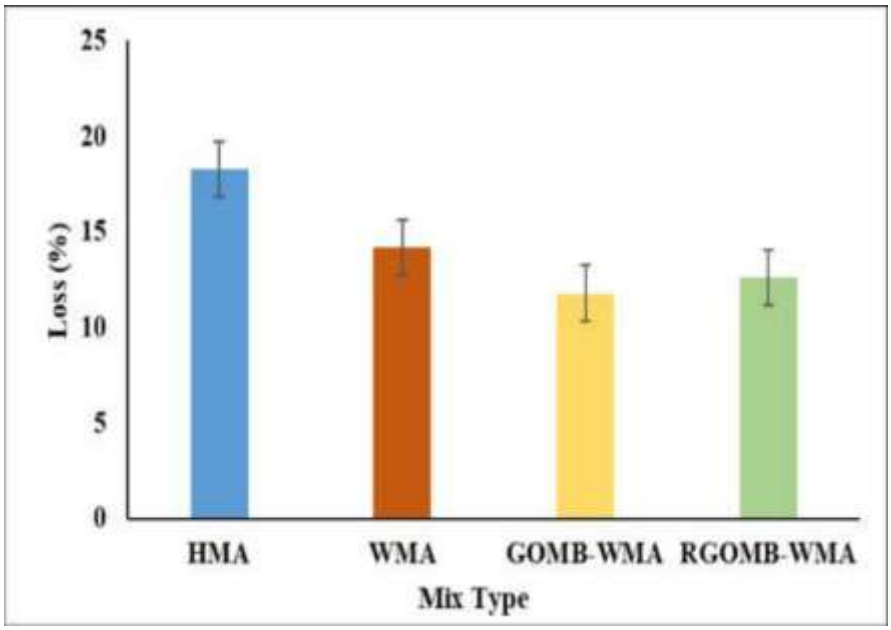



**Fig. 7.** Cantabro loss of HMA, WMA and 0.3% GO- and RGO-modified WMA

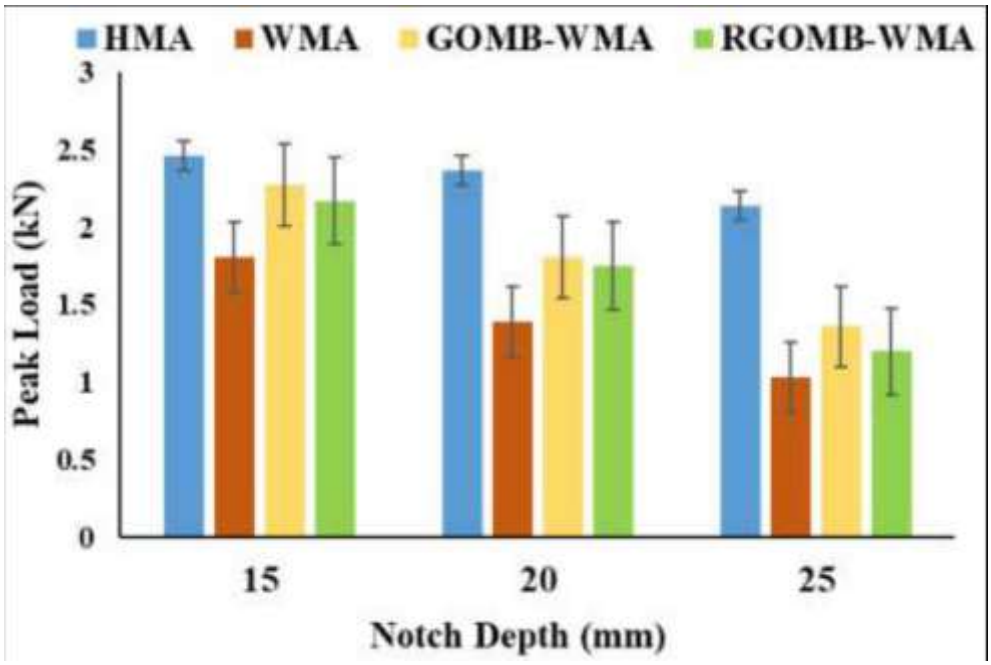



**Fig. 8.** SCB test results of HMA, WMA and GO- and RGO-modified WMA

## 5. Conclusions

1. GGBFS at 8% by weight of aggregate was the best filler for the DBM mix, giving an HMA stability of 14.04 kN, 27.28% and 13.39% higher than with stone dust and cement fillers.
2. Additive-free WMA can be produced by the two-phase mixing process; the mix prepared at 120 °C with an 80:20 bitumen-to-emulsion ratio was best, with a stability of 12.35 kN, a TSR of 88%, an RMS of 76.54% and a rut depth of 4.12 mm, close to HMA.
3. GO and RGO raised the softening point and reduced the penetration and ductility of bitumen in both unaged and PAV-aged states; 0.3% by weight of bitumen was optimum for the mixtures.
4. At 0.3%, GO- and RGO-modified WMA reached stabilities of 16.28 and 16.13 kN, about 31% above unmodified WMA and above HMA.
5. GO-modified WMA improved ITS to 680 kPa and TSR to 88.3%, reduced the rut depth at 50 °C from 5.52 to 3.91 mm, and reduced the ravelling loss to 11.8%, 35% below HMA; RGO gave the highest RMS among the WMA mixes (79.21%).
6. Graphene-modified WMA prepared by TPMP provides performance comparable to HMA at a production temperature 30–40 °C lower, offering an energy-efficient and durable alternative.

Field trials, fatigue and low-temperature cracking tests, and life-cycle cost and emission assessments are recommended before large-scale use.

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