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**Research Article**

# Exploring thermal behavior of recycled polymer-modified bitumen

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**ABSTRACT**

This study explores the modification of 60/70 grade bitumen using recycled low-density polyethylene (R-LDPE) polymer to enhance its thermal performance for flexible pavement. Rutting is a common form of distress in asphalt pavements, predominantly caused by the thermal behavior of bitumen, especially in areas with elevated temperatures. Four modified bitumen binders are prepared with varying R-LDPE content by weight (3%, 4%, 5%, and 6%). The effects of R-LDPE on bitumen are assessed through differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) to investigate the thermal behavior and stability of the modified binders. The primary objective of the research is to evaluate the dispersion of R-LDPE within the bitumen matrix and to conduct a comparative analysis of the thermo-mechanical properties of modified bitumen versus virgin bitumen. The results indicate that adding R-LDPE polymer improves the thermal performance of bitumen, demonstrating superior stability of flexible pavement for hot climates. Results have revealed the best rutting resistance for 6% R-LDPE.

**Keywords:** Polymer Modified Bitumen (PMB), Differential Scanning Calorimetry (DSC), Thermogravimetric Analysis (TGA), Asphalt, Flexible Pavement

## INTRODUCTION

Bitumen is not a new product for the world; evidence of its use dates back as early as 8100 BC. (1). For many years, bitumen has been used as a binder material in constructing flexible pavements. Although the quantity of bitumen used is significantly lower compared to aggregates by weight, it plays a critical role in influencing the performance of roads under varying temperatures and traffic loads (2,3). Bitumen is highly sensitive to temperature variations, causing its physical state to alter. It softens and becomes more fluid at elevated temperatures, while at lower temperatures, it hardens and turns brittle (4,5). When bitumen softens at elevated temperatures, it compromises the structural stability of pavements and may result in rutting failures. Conversely, at lower temperatures, it hardens, becoming a brittle and glass-like structure, leading to the thermal cracking of the road (6) (7). At present, numerous studies are actively being conducted on the modification of bitumen using recycled polymers, as it's not only effective as beneficial for waste polymers (8), but also improves the thermal stability and performance of asphalt (9). Also, the environmental impacts of plastic waste on human health are extremely severe, particularly in the second-most populated countries such as India (10). The proportion of LDPE (Low-Density Polyethylene) plastic waste constitutes a significant percentage when compared to other categories of plastic waste (11). Integrating R-LDPE into bitumen not only reduces polymer waste significantly but also enhances road durability through improved thermal performance (12). Indian Standard Code 15462:2004 classifies polymer and rubber-modified bitumen into the following four types:

- PMB(P) - Plastomeric thermoplastic-based
- PMB(E) - Elastomenc thermoplastic-based
- NRMB - Natural rubber and SBR latex-based
- CRMB - Crumb rubber/ treated crumb rubber based.

The scope of this research primarily focuses on Polymer-Modified Bitumen PMB (P). Bitumen consists of a mixture of asphaltenes and maltenes, making it a complex material (13). In this research, recycled low-density polyethylene (R-LDPE) polymer is used to modify bitumen thermal properties. Understanding the chemical characteristics of R-

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LDPE might be challenging because it is obtained from various sources, including thin polythene, bags, and other plastic items (14). R-LDPE's structural irregularities, coupled with the complex behavior of bitumen's maltenes and asphaltenes, further complicate a unified understanding of this polymer-modified mixture, necessitating comprehensive thermal and mechanical evaluations. Advanced analytical techniques such as Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) are effective for thermal stability analysis, which can provide better insights into the thermal analysis of polymer-modified bitumen (15–17). TGA helps to determine the thermal degradation behavior of PMB (18). DSC provides insights into phase transitions, glass transition temperature (19), and melting behavior, while TGA analyzes thermal stability, decomposition temperature, and oxidation resistance (20). These tests help determine the optimal polymer content and processing conditions, enabling the development of improved polymer-modified bitumen (PMB) for road construction.

## MATERIALS AND METHODS

### Material

Bitumen: The study utilized 60/70 grade bitumen sourced from the Mathura Refinery of Indian Oil Corporation Limited (IOCL). Properties of virgin bitumen were tested and listed in Table 1.

*R-LDPE:* A kind of commercially available recycled low-density polyethylene (R-LDPE) derived from post-consumer LDPE waste, processed to meet industrial standards, in granule form, is used for this investigation, i.e., the average particle size of 2-2.36 mm as determined by sieve analysis.

Table 1: Properties of virgin bitumen of grade 60/70

| Properties | Range | Result | Test Method |
|---|---|---|---|
| Penetration at 25°C | 60-70 | 66 | IS 1203 |
| Softening Point in °C | 47 min | 52 | IS 1205 |
| Flash Point in °C | 220 min | 236 | IS 1448 |
| Specific Gravity | 0.97-1.02 | 1.0 | IS 1202 |
| Ductility in cm | 50 min | 76 | IS 1208 |

*Preparation of samples:*

Initially, 60/70 grade bitumen was heated to a temperature range of 140°C to 150°C to transition it from a semi-solid (gel) state to a liquid state for workability. Simultaneously, recycled low-density polyethylene (R-LDPE) granules were heated to 150°C to 160°C to acquire a melted condition. The melted R-LDPE was then added to the hot liquid bitumen in proportions of 3%, 4%, 5%, and 6% by weight of the virgin bitumen. After combining both materials, the mixtures were subjected to thorough blending using a mechanical stirrer operating at 8000 rpm for one hour to produce polymer-modified bitumen samples. After the end of the mixing process, the samples were removed from the flask and divided into small containers, covered with caps, and stored for the testing program. TGA and DSC tests are performed after two weeks of storage of samples for this study to simulate extended service conditions, and bitumen-polymer compatibility over time, allowing time for oxidation and further aging effects to develop.

The binders used in this study were coded as below;
Virgin bitumen– "B";
Virgin bitumen + 3% R-LDPE– "PMB3";
Virgin bitumen + 4% R-LDPE– "PMB4";
Virgin bitumen + 5% R-LDPE– "PMB5";
Virgin bitumen + 6% R-LDPE– "PMB6";
*Testing Program:*

Conventional tests: Penetration test, softening point, ductility, and flash point tests were carried out on both virgin and R-LDPE modified bitumen samples of different percentages by weight.

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### TGA analysis

Thermogravimetric analysis (TGA) is a straightforward thermal analysis technique that involves measuring a sample's weight change as it is heated. This method provides valuable insights into the decomposition of materials, thermal behavior, and their reactions with surrounding gases during heating (21). The results from a TGA experiment are typically presented as a curve that plots weight change against temperature for both virgin bitumen and different polymer-modified bitumen samples. The temperature is plotted on the horizontal axis in degrees Celsius, while the weight change is shown on the vertical axis as a percentage. The temperatures corresponding to the start and end of each thermal event are influenced by testing conditions, including the heating rate, ambient oven conditions, and the characteristics and shape of the sample. As the heating rate increases, these temperatures tend to shift to higher values, leading to a delay in achieving thermal equilibrium. The weight change during heating is influenced by the material's properties and occurs within a specific temperature range. Thermogravimetric analysis (TGA) is performed on the PerkinElmer TGA 4000 Instrument. Sample pellets with masses ranging from 10 to 20 mg were heated at 10°C per minute from 30°C up to 600°C.

### DSC analysis

A Differential Scanning Calorimeter (DSC) is a highly effective instrument used to measure changes in the heat capacity of materials. It is employed for analyzing the thermal behavior of materials by detecting variations in their heat absorption or release as a function of temperature. It identifies endothermic or exothermic reactions that take place when a material undergoes a phase change, such as from solid to liquid. By studying the changes in the material's heat transfer properties during these phase transitions, such as solid-to-liquid transformations, the DSC provides detailed insights into thermal behavior. A Perkin-Elmer differential scanning calorimeter, model DSC 4000, is used for thermal determinations. The standard aluminum crucible is used as a reference. Nitrogen gas at 20 ml/minute is passed over the sample holder during the experiment. The sample weights for each DSC run ranged from 5 mg to 15 mg. DSC tests are conducted under a heating range from 35°C to 400°C at a heating rate of 20°C/min.

## RESULTS AND DISCUSSION

Samples were subjected to a variety of conventional testing, such as penetration, softening point, ductility, and flash point tests. Results of testing for PMB3, PMB4, PMB5, and PMB6 were compared with virgin bitumen and also with each other, respectively.

- Penetration by Grade

A penetration test is an indirect test to determine the consistency and hardness of bitumen samples. Readings are measured with a 5-second standard penetrometer of Aimil (1 division of 0.1 mm) at 25°C for this research. The basic concept of this test is harder the bitumen, the lower be penetration depth of the needle (22). Fig. 1 indicates that when LDPE polymer content rises from 3% to 6%, the penetration value decreases from 58.7 mm to 46.3 mm. The data clearly show that adding LDPE polymer causes an increase in bitumen's brittleness. The bitumen is harder when the penetration values are lower. The greater resistance to rutting for flexible pavement may be attributed to the increased hardness.

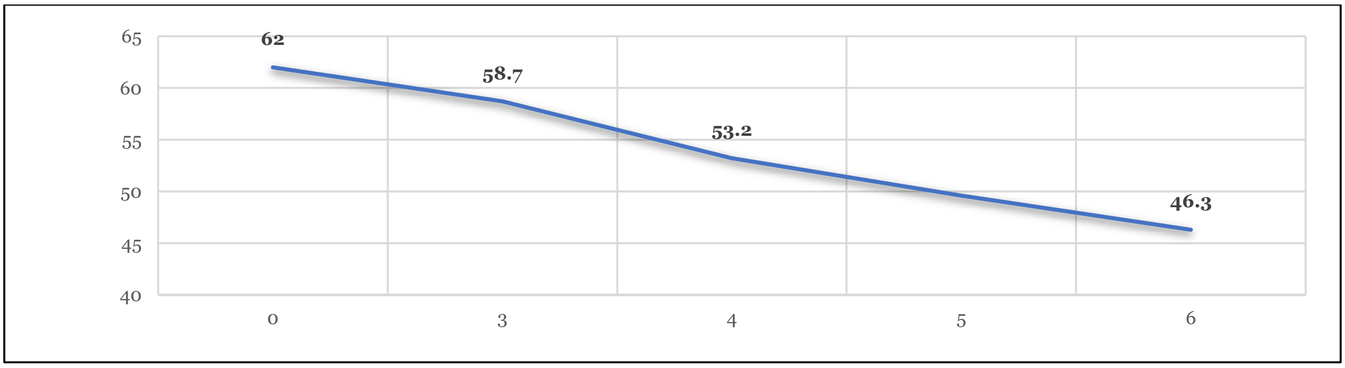


Fig. 1 Effect of R-LDPE polymer on penetration values

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- Softening Point

The softening point provides details regarding the temperature at which bitumen begins to soften. Bitumen with higher softening points is more resistant to temperature changes(23). The Ring and Ball apparatus is used as per IS:1205-1978 specification for this research.

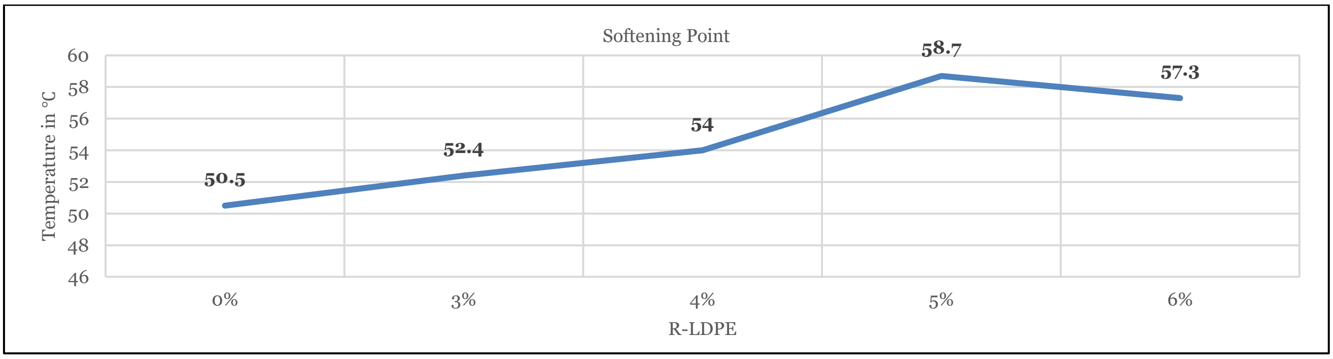


Fig. 2 Results of the softening point of different % of PMB

The results of the softening point test performed on PMB3, PMB4, PMB5, and PMB6 samples are tabulated in Fig. 2, which shows the increase in softening point from 52.4°C of PMB3 to 58.7°C of PMB5. A higher softening point simply means higher heat resistance and softens at comparatively higher temperatures. It will reduce the rutting failures and pavement unevenness in bituminous roads, especially in summer.

- Ductility

Ductility is the property of materials to stretch, which is crucial for evaluating the performance of bitumen in flexible pavement construction. These roads experience diverse stresses like changes in temperature, heavy traffic loads, and lateral base movements, requiring bitumen to have good ductility to resist cracking. By conducting a ductility test, it could be confirmed that bitumen meets the required standards for durability and better performance in all weather conditions. Ductility Testing Machine (AIM 565-1) is used at 25°C as per IS:1208-1978 specification for this research. Three briquette molds were tested for each sample of PMB3, PMB4, PMB5, and PMB6. It has been observed that with the addition of polymers, the stiffness of bitumen increases simultaneously ductility of PMB is decreased (24,25).

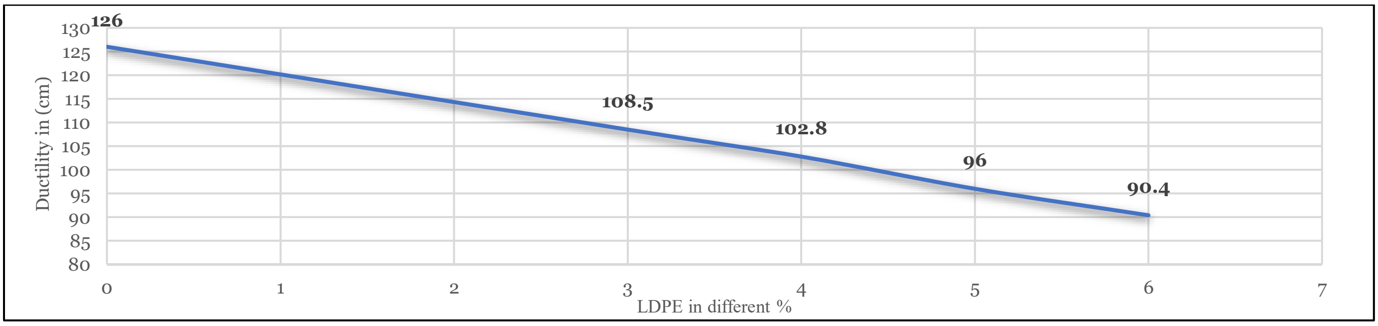


Fig. 3 Effect of R-LDPE on ductility values

Reduction in ductility could be because of the interlocking of LDPE granules with bitumen. Figure 3 depicts a drop in ductility from 108.5 cm to 90.4 cm as the proportion of LDPE increased from 3% to 6%. LDPE modification makes bitumen more brittle, resulting in the length of the thin wire in the ductility test continuously decreasing as the percentage of LDPE modifiers is increased.

- Flash Point

At ambient temperatures, bitumen exists in a semi-solid state and must be heated to attain fluidity suitable for mixing. Since it is a petroleum by-product, it poses a fire hazard and requires careful handling during the heating

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process. To mitigate fire risks, strict adherence to temperature control protocols is imperative(26). Safety measures encompass specialized equipment designed for controlled heating and monitoring of bitumen temperatures to ensure they remain below the specified fire point.

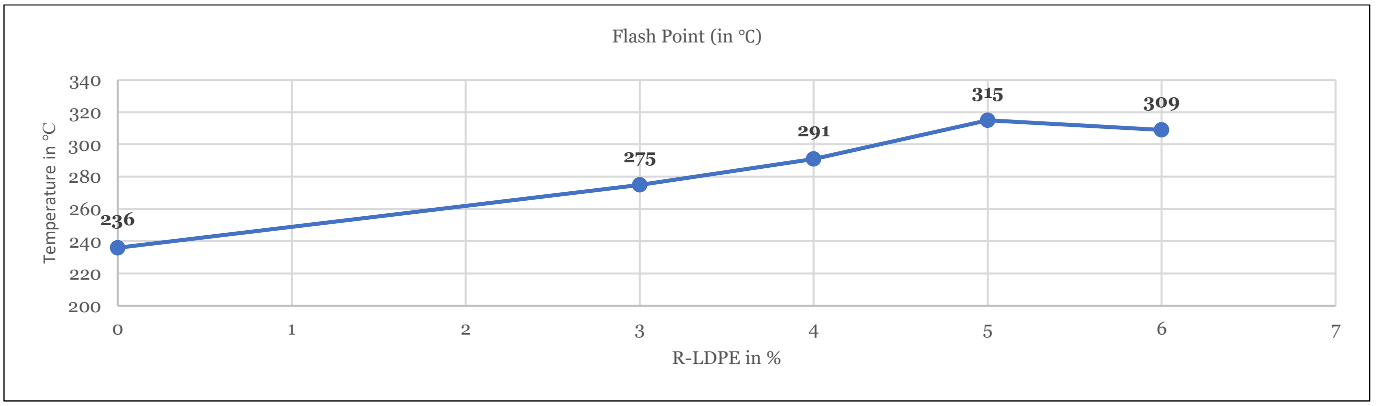


Fig. 4 Flash Point of different PMB

The open cup cleave land apparatus is used as per IS:1448-69 (1969) for this research. Figure 4 displays the flash point of bitumen modified with 0%, 3%, 4%, 5%, and 6% R-LDPE granules.

- Thermogravimetric Analysis

The Thermogravimetric Analysis (TGA) conducted on R-LDPE modified bitumen offers a comprehensive understanding of its thermal behavior, which is crucial for assessing its suitability in road applications.

The TGA data reveal that R-LDPE modified bitumen expresses improvement in thermal stability compared to unmodified bitumen. The weight loss profile is more gradual, suggesting that R-LDPE helps maintain the integrity of the bitumen over extended periods of thermal stress, which is important for its long-term durability.

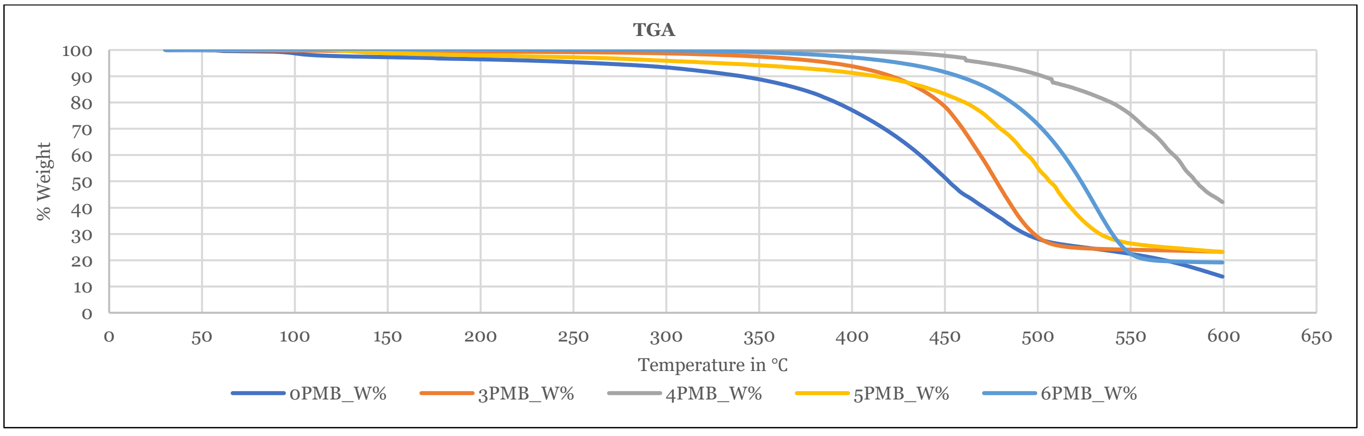


Fig. 5 Thermogravimetric Analysis of Different PMB

The modification effectively delays the onset of degradation, indicating an enhanced resistance to high-temperature conditions typically encountered during the service life of roads.

Key observations of the TGA curve from Fig. 5 for different R-LDPE modified bitumen samples are as follows:

Thermal Stability Trends:

All samples start at 100% weight retention, confirming that no initial decomposition occurs. As temperature increases, weight loss occurs due to the degradation of bitumen and polymer additives.

Effect of R-LDPE Content:

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The degradation pattern shifts with increasing R-LDPE content. Higher R-LDPE content (PMB5, PMB6) shows better thermal stability initially, but undergoes decomposition at higher temperatures.

Decomposition Stages:

Bitumen generally exhibits a multi-stage decomposition process, with polymer-modified samples showing a delayed onset of weight loss, indicating enhanced thermal resistance

All tested samples, including pure bitumen (0% PMB), remained stable up to approximately 250°C, showing minimal weight loss at lower temperatures. However, as the polymer content increased, the onset of decomposition shifted to higher temperatures, indicating improved thermal resistance. Unlike un-modified bitumen, which exhibited a single significant degradation phase, polymer-modified samples (3%, 4%, 5%, and 6% PMB) experienced a more gradual weight reduction, suggesting multiple breakdown stages due to the polymer's influence. These results confirm that incorporating polymers into bitumen improves its resistance to high temperatures, making it more suitable for applications like road construction in hot climates.

- Differential Scanning Calorimetry

Glass Transition Temperature

The glass transition temperature represents the point where the material transitions from a rigid, glassy state to a more rubbery, flexible state. The DSC curves show variations in Tg across different PMB compositions, indicating that polymer modification alters the bitumen's thermal behavior. Typically, increasing polymer content raises the Tg, suggesting improved resistance to low-temperature cracking.

Melting and Softening Behavior

Around 100°C, an endothermic peak is observed, which may correspond to the softening or phase transition of bitumen. The enthalpy change in this region indicates the amount of heat energy absorbed during the phase transition. Higher PMB concentrations broaden this transition, implying better thermal stability and reduced susceptibility to softening under high temperatures.

Crystallization of Polymer Domains

At lower temperatures, polymer-modified bitumen may exhibit exothermic crystallization peaks, depending on the polymer type and its dispersion in bitumen. The enthalpy associated with crystallization provides insights into how polymers interact with bitumen molecules. Higher polymer content (PMB6) might show distinct double peaks, indicating phase separation or incomplete miscibility.

**DSC Results:** The DSC thermograms indicate that the inclusion of R-LDPE alters the phase transition characteristics of the bitumen. A shift in the glass transition temperature (Tg) and changes in the melting behavior are observed, indicating that R-LDPE modifies the bitumen's molecular structure, improving its thermal response. These changes enhance the bitumen's flow properties and flexibility, making it better suited for varying temperature conditions in road environments. The thermal properties of the modified PMBs obtained from Fig. 6 showed that the R-LDPE modified bitumen is stiffer than virgin bitumen and provides better stability of the road at high temperatures.

Effect of Polymer Content on Enthalpy

- 0% PMB (Virgin Bitumen): Shows a relatively sharp transition with lower enthalpy changes. Exhibits a more defined melting peak, indicating crystalline regions in the bitumen structure.
- PMB3 and PMB4: Moderate polymer content reduces the crystallinity of bitumen, leading to smoother enthalpy transitions. The melting enthalpy decreases slightly, indicating improved thermal stability.
- 5% PMB (Optimal Composition): Displays a well-distributed enthalpy change with stable heat flow characteristics. Suggests uniform polymer dispersion, preventing abrupt thermal transitions. Lower crystallization enthalpy means reduced segregation of polymer and bitumen phases.

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- 6% PMB: The DSC curve suggests higher thermal stability but possible phase separation at elevated temperatures. A secondary peak in the heat flow may indicate polymer-rich domains that retain their structure at high temperatures. Higher enthalpy values could imply increased energy absorption before degradation.

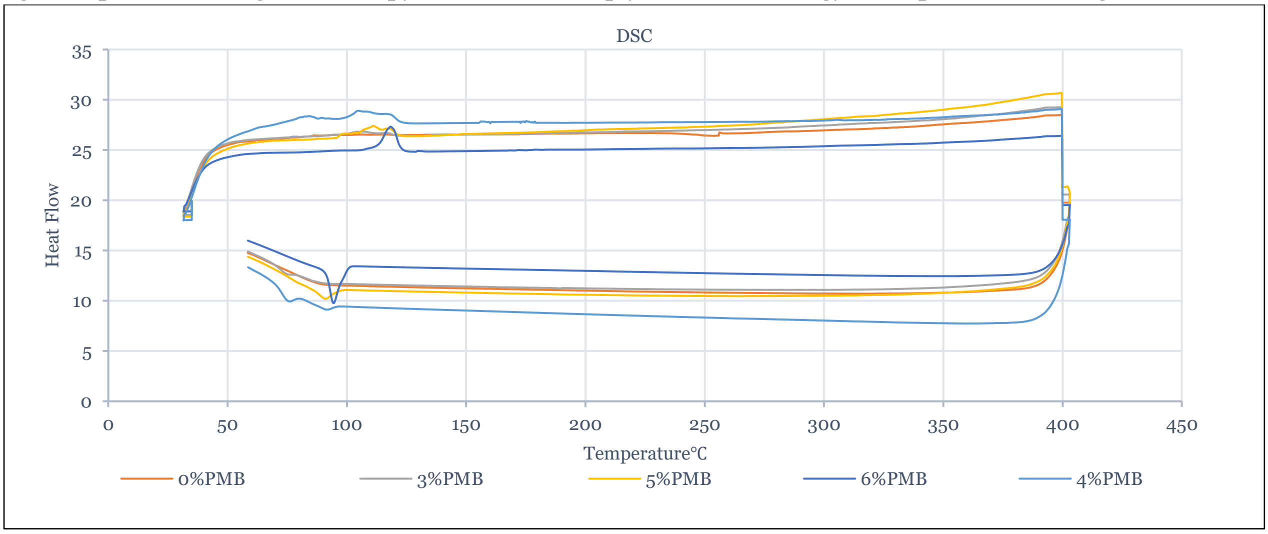


Fig. 6 DSC analysis of different PMB

## CONCLUSION

The following conclusions can be drawn based on the results obtained in the study.

**Observations from the Conventional tests:**

- Penetration Value: Increasing the concentration of R-LDPE (3%, 4%, 5%, and 6%) results in a decrease in the penetration value of modified bitumen, signifying greater brittleness as the polymer content rises.
- Softening Point: Bitumen modified with 5% R-LDPE (PMB5) exhibits the highest softening point at 58.7°C, which is approximately 8°C above that of the virgin bitumen. This indicates enhanced resistance to temperature variations.
- Ductility: As R-LDPE concentration increases, ductility decreases, which suggests improved resistance to rutting, making the modified bitumen suitable for tropical climates.
- Flash Point: PMB5 shows the highest flash point, confirming that incorporating polymers into bitumen improves enthalpy.

**Observations from the TGA Graph:**

- 0% PMB (Virgin bitumen) starts degrading at a lower temperature, indicating lower thermal stability.
- PMB3 and PMB4 improve thermal resistance but still show significant weight loss earlier than higher percentages.
- PMB5 and PMB6 show the highest onset temperature and slower degradation, meaning they provide better thermal resistance.
- PMB6 appears to provide the best thermal stability, as it degrades at the highest temperature and retains more weight at 600°C.

**Observations from the DSC Graph:**

The DSC analysis shows that polymer modification significantly influences the enthalpy changes of bitumen. The 5% PMB composition appears to be optimal, balancing thermal stability and phase compatibility. Higher polymer contents (PMB6) may lead to phase separation, while lower contents (3–4% PMB) offer moderate improvements over unmodified bitumen. These findings highlight the role of enthalpy in assessing the effectiveness of polymer-modified bitumen for pavement applications.

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*Glass Transition and Melting Behavior:*

The curves indicate different thermal transitions, with notable changes in heat flow around 100°C and at higher temperatures.

*Effect of Polymer Content:*

Increasing polymer content affects the thermal stability and heat flow. The PMB5 sample appears to have a relatively stable profile, suggesting optimal dispersion of the polymer.

*Crystallinity and Thermal Stability:*

Higher polymer content (e.g., PMB6) shows a slightly different heat flow pattern, which might indi-cate phase separation or higher thermal stability.

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