

INVESTIGATING THE CHEMICAL PROPERTIES, THERMAL AND MICROSTRUCTURAL CHARACTERIZATIONS OF INDUSTRIAL WASTE BASED ASPHALT CONCRETE FOR ENHANCED PERFORMANCE

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Abstract

This research addresses the growing need for sustainable alternatives in road construction by investigating the use of industrial waste materials specifically steel slag and fly ash as partial replacements for natural aggregates in asphalt concrete. The problem lies in the environmental impact of quarrying natural aggregates and the under-utilization of industrial by-products, which pose disposal challenges. To explore this, steel slag and fly ash were incorporated into asphalt mixtures at varying replacement levels (2%, 4%, 6%, and 8%) to assess their suitability as substitutes. The methodology involved characterizing the materials using physical tests (Aggregate Impact Value, Aggregate Crushing Value, and penetration tests), as well as chemical and thermal analyses (X-ray diffraction, X-ray fluorescence, and thermogravimetric analysis). The mechanical properties of the modified asphalt were evaluated through Marshall Stability and flow tests to measure strength and deformation under load. Results show that the inclusion of steel slag and fly ash significantly enhanced the stability and durability of asphalt concrete, with optimal performance observed at 6% replacement. This study concludes that these industrial waste materials can serve as sustainable alternatives in asphalt production, reducing environmental impacts while meeting road construction standards in Nigeria.

1.0 INTRODUCTION

The resilience and longevity of road pavements are key concerns in highway engineering due to challenges such as climate variation, heavy traffic loads, cracking, asphalt aging, moisture infiltration, and environmental impacts. To address these issues, modifying asphalt constituents has emerged as an effective solution. A promising approach is the incorporation of industrial waste, such as steel slag and fly ash, into asphalt mixtures, which enhances performance and promotes environmental sustainability. The practice addresses waste disposal concerns and improves asphalt properties, offering a sustainable option for road construction [1], [2].

Steel slag, a byproduct of steel manufacturing, has been widely used in asphalt concrete for its ability to enhance strength, durability, and resistance to fatigue and rutting. The angularity and rough texture of steel

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slag particles improve bonding and interlocking in asphalt mixtures [3]. A study [4] reported higher Marshall Stability and indirect tensile strength in steel slag asphalt mixtures compared to conventional ones. Additionally, using steel slag conserves natural resources by reducing the need for virgin aggregates [5]. Fly ash, a byproduct of coal combustion, acts as a mineral filler, reducing void content and increasing cohesiveness in asphalt mixtures. Research has demonstrated that asphalt blends containing fly ash show better resistance to thermal cracking and moisture damage [6]. Fly ash incorporation has been found to improve mechanical properties and performance, particularly at 8% replacement, achieving a Marshall Stability value of 8.22 kN, suitable for heavy traffic and aligning with Nigerian road specifications [7].

From an environmental perspective, incorporating steel slag and fly ash in asphalt offers several advantages, including recycling industrial waste, reducing the carbon footprint of road projects, and minimizing landfill waste [8]. Economically, these materials provide a cost-effective alternative to traditional construction materials, reducing expenses while fostering sustainable development [9]. Case studies have confirmed the effectiveness of these materials in improving pavement performance. In Ohio, for example, steel slag incorporation improved load-bearing capacity and extended pavement lifespan [10]. Similarly, in Nigeria, steel slag asphalt concrete at 8% replacement achieved optimal stability for heavy-traffic roads [11].

Hybrid modifications, involving combinations of steel slag, fly ash, and polypropylene, have also been explored. These combinations have been shown to significantly enhance Marshall Stability and flow properties, with maximum stability values of 13.50 kN achieved at 8% replacement [12].

The process of modifying asphalt involves adding materials to traditional constituents to improve properties like strength, durability, and resistance to rutting, cracking, and aging. Advanced characterization techniques such as X-Ray Fluorescence (XRF), X-Ray Diffraction (XRD), Thermogravimetric Analysis (TGA), and Scanning Electron Microscopy (SEM) are used to understand the molecular dynamics and micro-scale interactions in modified asphalt [13], [14]. XRD studies of steel slag reveal crystalline phases like tricalcium silicate (C3S) and dicalcium silicate (C2S), which enhance the bonding properties and load-bearing capacity of asphalt mixtures [15]. Fly ash contains alumina and amorphous silica, with crystalline phases such as mullite and quartz, which contribute to its pozzolanic

activity, improving long-term strength in asphalt mixtures [16].

2.0 MATERIALS AND METHODS

2.1 Work Materials

The materials used in this study included 60/70 penetration grade bitumen, aggregates, and fillers, which were sourced from Slava Yeditepe Construction Company Limited, located in Osogbo, Nigeria. Steel slag, a byproduct of the steel manufacturing process, was obtained from the Ife Steel and Iron Company in Ile-Ife, Osun State, Nigeria. Steel slag forms when molten steel is separated from impurities in steel-making furnaces, as depicted in Figure 1. Additionally, fly ash, a fine, gray, glassy powder produced as a byproduct in coal-fired power plants, was acquired from burning chambers near the thermal power station in Oji River, Enugu State, Nigeria (Figure 2).

2.2 Experimental Methods

The selected aggregates, steel slag, and fly ash were sun-dried before being subjected to mechanical sieve analysis to assess their particle size distribution. Various tests were conducted to determine the physical properties of the materials, including the moisture content test [17], Aggregate Impact Value (AIV) test [18], Aggregate Crushing Value (ACV) test [19], specific gravity test [20], and flakiness and elongation tests [21]. The bitumen underwent a series of tests to determine its quality, including penetration test [22], softening point test [23], flash and fire point test [24], ductility test [25], and viscosity test [26]. The chemical properties of the steel slag and fly ash were analyzed using X-ray diffraction [27], X-ray fluorescence [28], and thermogravimetric analysis [29], [30].

For the asphalt concrete mix, steel slag (SS) and fly ash (FA) were each incorporated separately at 2%, 4%, 6%, and 8% to partially replace conventional aggregate and filler materials. The asphalt concrete was prepared with a composition of 56% fine aggregate, 39% coarse aggregate, and 5% (60 g) mineral filler. Five different bitumen contents were used: 5.0%, 5.5%, 6.0%, 6.5%, and 7.0% of the total aggregate mass. The optimum bitumen content was determined through trial testing.

The asphalt concrete samples were compacted in cylindrical molds using a 4.5 kg rammer, with 50 to 75 blows applied (Figure 3). The final modified asphalt concrete samples were produced (Figure 4), and their Marshall Stability and Flow were evaluated.





Figure 1: Steel Slag



Figure 2: Fly Ash



Figure 3: Sample of asphalt concrete in compaction mould



Figure 4: Asphalt Concrete Samples Produced

3.0 RESULTS AND DISCUSSION

3.1 Sieve Analysis (BS 812-103, 1990)

Figure 5 provides the aggregates' sieve analysis result. It is evident from Figure 5 that the selected aggregate fall within the standard specific envelope for the design of a hot asphalt mix. The Coefficient of Gradation (CC) and Uniformity coefficient (CU) can be calculated from the particle distribution curves in Figure 5 using Equations (1) and (2) [31]

$$\text{Uniformity coefficient (CU)} = \frac{D_{60}}{D_{10}}, \quad (1)$$

From Figure 1

$$D_{60} = 0.5, D_{30} = 0.3, \text{ and } D_{10} = 0.15$$

To determine the uniformity coefficient 'CU', from Equation 1

$$C_u = \frac{D_{60}}{D_{10}} = 3.333$$

Coefficient of gradation C_c is given by

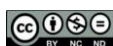
$$C_c = \frac{D_{30}^2}{D_{60} \times D_{10}} \quad (2)$$

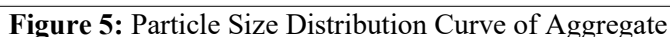
$$C_c = \frac{D_{30}^2}{D_{60} \times D_{10}} = \frac{0.3^2}{0.5 \times 0.15} = 1.2$$

The aggregate is categorized as well-graded aggregate based on its coefficient of gradation (C_c), which ranges from 1 to 3. The aggregate sample's sizes vary from coarse to fine to allow for interlocking between them. The outcomes are within the parameters given in the Federal Ministry of Work Highway Manual [32].

3.2 Test Result of Physical Properties of Aggregates, Steel Slag and Fly Ash

The outcome of the result, which is displayed in Table 1, disclosed the characteristics of the steel slag, aggregates and fly ash that were utilized. The values found for each parameter attribute are within the allowed range for road paving [32]. Additionally, it is evident that the moisture content (M.C), AIV, and ACV values of the steel slag were lower than those of the aggregates. This indicates that steel slag possesses slightly better mechanical properties, making it more suitable for enhancing asphalt mix performance. Fly ash, with its lower specific gravity, provides filler benefits that improve workability and reduce mix density.



**Table 1:** Summary of physical properties of Aggregates, steel slag, and fly ash

S/N	Materials	M.C(%)	AIV (%)	ACV (%)	Gs (g)	Flakiness and Elongation Index Test (%)
1	Aggregate	0.95	25.20	33.98	3.09	22.64
2	Steel Slag	0.56	23.60	31.96	3.12	27.41
3	Fly Ash	1.16	--	--	2.31	--
	[32]	Max 5%	20-30	27.30	--	Max 30

3.3 Test Result of Physical Properties of Bitumen

Table 2 summarizes the bitumen test results, showing that all measured values, including penetration, softening point, flash point, fire point, ductility, viscosity, and water content, fall within the acceptable range specified by the Federal Ministry of Works Highway Manual [32]. These results confirm the quality of the bitumen for use in asphalt mix design. The high ductility value indicates good flexibility, while adequate softening and flash points ensure resistance to deformation and thermal stability during service.

3.4 Test Result of Chemical and Mineralogy Characterization of Steel Slag

The X-ray diffraction (XRD) results in Figure 6 reveal that steel slag mainly comprises SiO_2 , with minor amounts of K_2O , MgO , CaO , and Al_2O_3 . Crystalline phases like C_2S , C_3S , and C_4AF were identified,

confirming the crystalline material characteristics, as supported by previous studies [33][34][35][36][37].

Table 2: The Physical Characterization of Bitumen

FMWHM, 2013 [32]	Properties	Obtained Value
Min.75	Ductility,cm	150
Min 200	Flash Point °C	210
Min.240	Fire Point °C	255
Min.100	Viscosity, sce	137
Min 3	Water-in-Bitumen 3.4	4.5
60-70	Penetration, mm	64
Min. 45	Softening Point °C	48

XRF analysis (Table 3) corroborates this composition. The high Ca and Fe content further supports its suitability for structural stability and strength enhancement in pavement applications.

The XRD patterns for steel slag and fly ash revealed distinct differences in their crystalline and amorphous phases. Steel slag exhibited prominent peaks corresponding to crystalline minerals such as quartz (SiO_2), hematite (Fe_2O_3), and magnetite (Fe_3O_4), which are associated with high hardness and abrasion resistance, making the material suitable for use in high-stress pavement layers. The presence of calcium silicates also suggests good potential for chemical bonding with asphalt binder, enhancing mixture stability.

Fly ash, in contrast, showed a predominantly amorphous hump with minor crystalline peaks, indicating high amorphous silica and alumina content. This amorphous silica is reactive and can improve binder-aggregate adhesion through pozzolanic interactions, contributing to better resistance against stripping and moisture damage in asphalt mixes. The combination of crystalline phases in steel slag and the amorphous phases in fly ash provides complementary benefits — increased mechanical strength from steel slag and improved binder compatibility from fly ash — thereby justifying their joint consideration as partial replacements for conventional aggregates and fillers in asphalt concrete production.

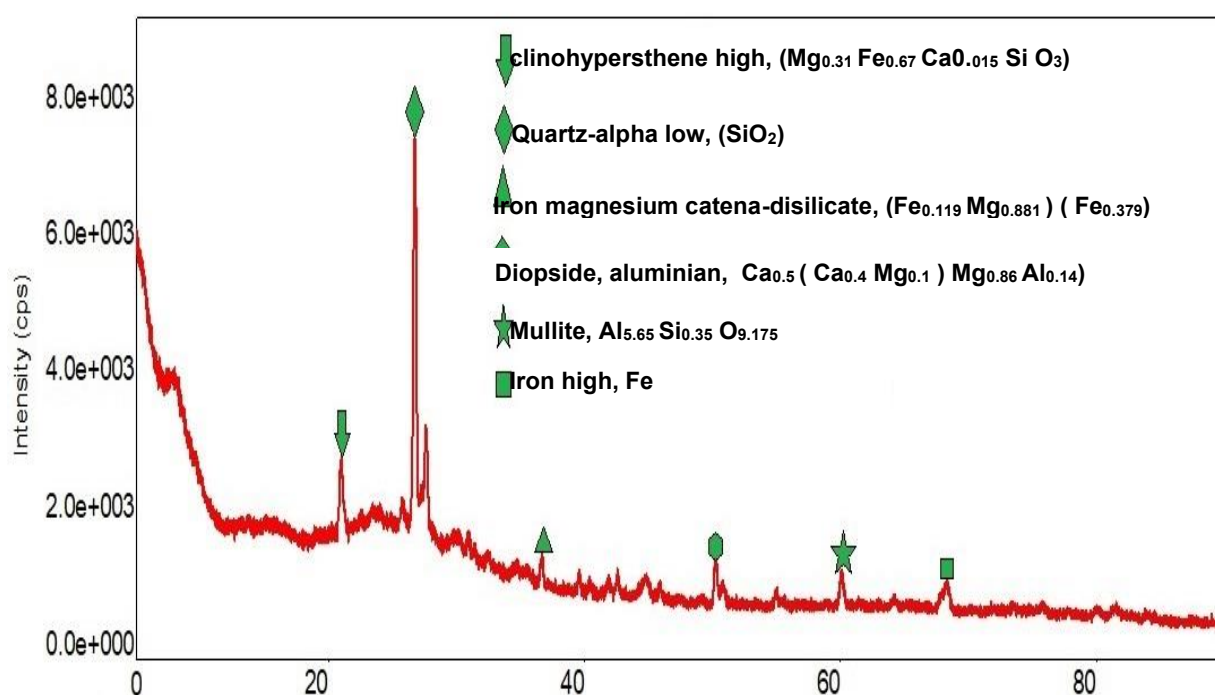


Figure 6: X-ray Diffractogram of Steel slag

Table 3: Mass Composition of Main Elements in the Steel Slag

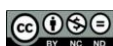
Element	Ca	Fe	Si	Mg	Al	K	Na
Concentration	60.83	17.10	11.80	5.66	2.04	1.49	1.08

3.5 Thermal Characterization of Steel Slag

The result of thermogravimetric analysis of steel slag showed three endothermic peaks in Figure 7. The first endothermic peak is recorded between 50-100°C with

weight loss of 0.0125%, attributed to dehydration of calcium silicate hydrate (C-S-H).

The second peak was observed at 350°C with weight loss of 0.001%, due to dehydration of calcium hydroxide (CH). The last endothermic peak appeared between 650-750°C with weight loss of 0.02%, linked to decarbonation of calcium carbonate (CaCO_3). These transitions indicate the slag's stable thermal behavior across typical pavement temperature ranges. The decomposition peaks also indicate stability under asphalt production temperatures, with minimal degradation below 500°C.



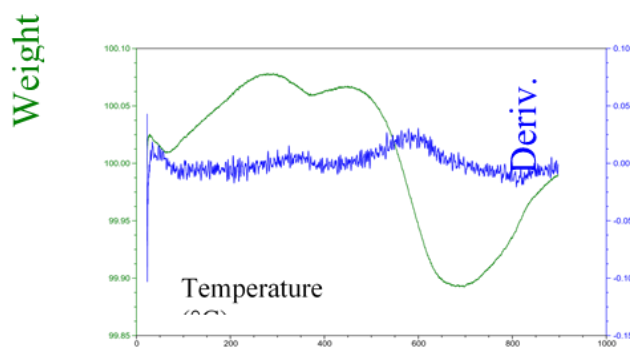


Figure 7: Thermo gravimetric – Differential Scanning Calorimeter Pattern of Steel

3.6 Test Result of Chemical and Mineralogy Characterization of Fly Ash

The XRD analysis of fly ash in Figure 8 shows a composition mainly of alumina and amorphous silica, with crystalline phases including quartz, hematite, and mullite, as well as traces of calcite and mica [38], [39]. Identified polymorphs include aluminum iron silicide and mica phases such as phlogopite, muscovite and biotite [40], [41]. XRF analysis indicates that fly ash contains 66.32% silica, consistent with previous reports [38], [42]. This high silica content enhances pozzolanic activity, improving the durability and long-term strength of asphalt mixtures.

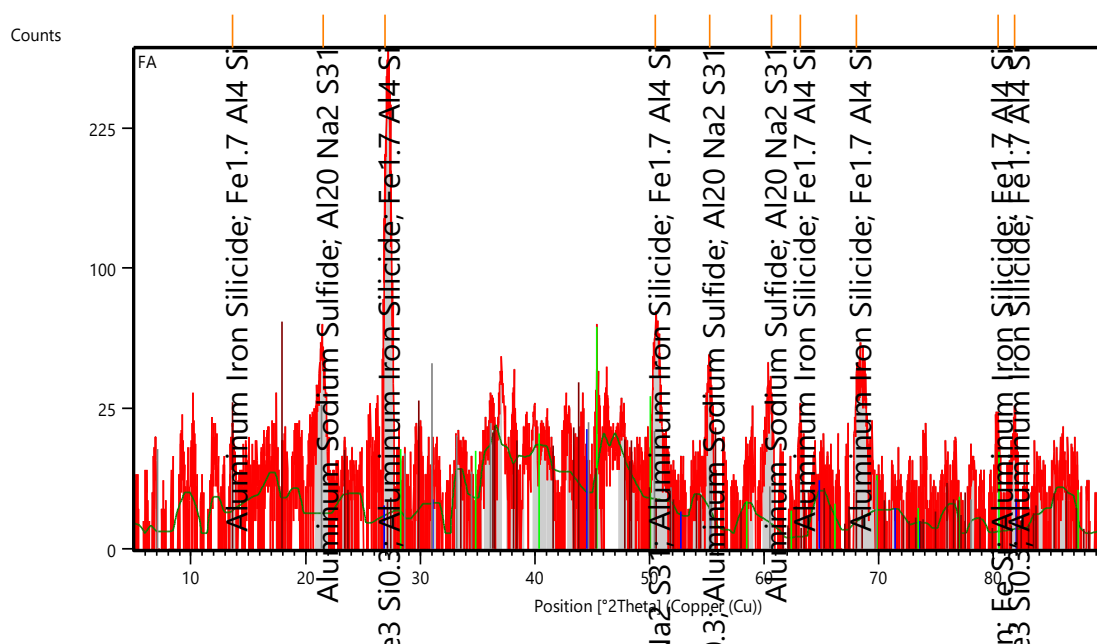


Figure 8: X-Ray Diffractogram of Fly Ash

Table 4: Mass Composition of Main Elements in the Fly Ash

Element	Si	Al	Fe	S	Ti	Ca	K
Concentration	61.32	25.29	4.44	3.02	2.35	1.98	1.60

3.7 Thermal Characterization of Fly Ash

Figure 9 shows two distinct steps of thermal degradation in the fly ash sample. The first degradation, occurring between 80 and 120 °C, resulted in a weight loss of 0.875%, primarily due to the evaporation of physically bound moisture (H₂O).

The second degradation, observed between 780 and 820 °C with a weight loss of 0.35%, is attributed to the dehydroxylation of calcium hydroxide [Ca(OH)₂] and the decomposition of carbonates (CO and CO₂). These results indicate that fly ash remains thermally stable within the typical operational temperature range of asphalt pavements (generally below 80 °C under service conditions and up to about 160 °C during mixing). The minimal mass loss at higher temperatures also suggests that its mineralogical composition remains largely intact during asphalt production, ensuring consistent performance as a filler material. This high-temperature resistance further supports its suitability for use in warm and hot-mix asphalt applications, where thermal degradation could otherwise compromise mechanical integrity.

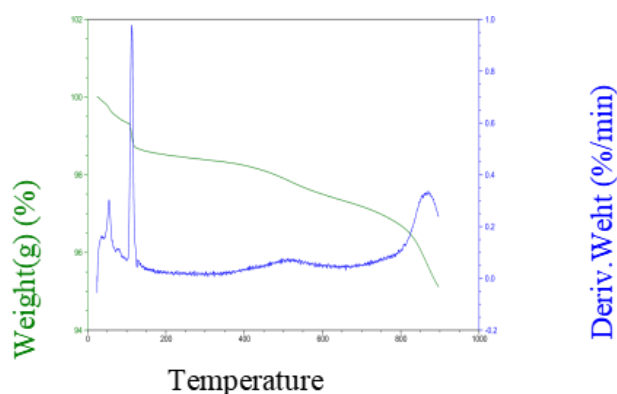


Figure 9: Thermogravimetric-Differential Scanning Calorimeter Pattern of Fly Ash

3.8 Marshal Stability Result of Steel Slag Modified Asphalt

Figure 10 illustrates the stability values for different bitumen contents used in this study. The highest stability of 13.00 kN was achieved at 6% bitumen content with 8% steel slag. Other optimum stability values include 11.50 kN at 6% steel slag, 12.20 kN at 4%, and 10.80 kN at 2%. All these values conform to the Nigeria General Specification for Roads and Bridges [43], which requires a minimum stability of 3.5 kN. These results indicate that the incorporation of steel slag improves structural resistance, which is crucial for heavily trafficked pavements. The test was chosen due to its proven relevance in predicting field performance and deformation resistance of asphalt mixture.

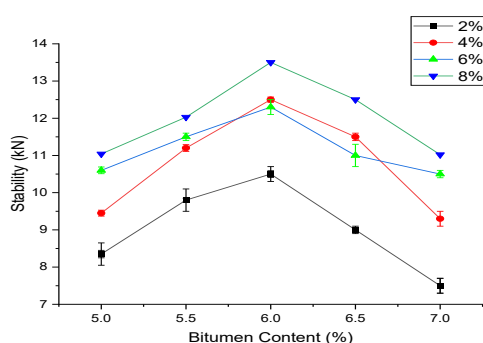


Figure 10: Marshal Stability of Steel Slag Modified Asphalt against Bitumen Content

Figure 11 shows the flow values for different bitumen contents. The highest flow value of 3.3 mm was recorded at 6.0% bitumen content with 2% steel slag. Other notable flow values include 3.10 mm at 4%, 3.00 mm at 6%, and 2.80 mm at 8% steel slag. These flow values comply with the NGSRB specification [43], which sets the acceptable range for flow between

2 mm and 6 mm. Notably, the results highlight that increasing slag content beyond 6% does not significantly improve flexibility, suggesting an optimum replacement range for field application.

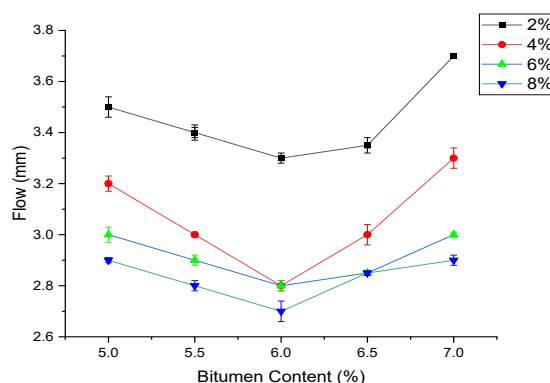


Figure 11: Marshal Flow of Steel Slag Modified Asphalt against Bitumen Content

3.9 Marshal Stability Result of Fly Ash Modified Asphalt

The stability results for fly ash-modified asphalt in Figure 12 show an initial increase in stability with rising bitumen content, peaking at 10.6 kN for 8% fly ash. The optimal bitumen contents were 5.5% for 2% and 4% fly ash, and 6.0% for 6% and 8%. These findings suggest that 2% to 8% fly ash replacements are effective for various traffic conditions, adhering to the Marshall Design criteria [44]. The improvement in stability is linked to the filler effect of fly ash particles, which enhance interlocking and load distribution.

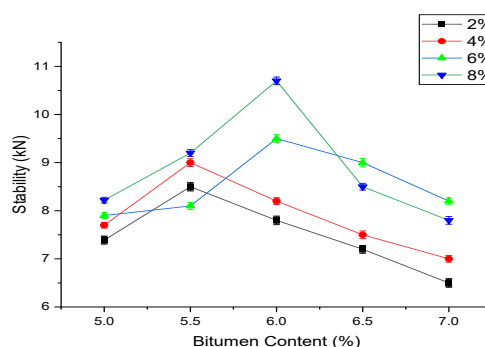


Figure 12: Marshal Stability of Fly ash Modified Asphalt against Bitumen Content

Figure 12 shows the flow values of fly ash-modified asphalt. An initial decrease in flow value was observed with increasing bitumen content, followed by a rise at higher percentages. The optimum flow values of 3.50 mm, 3.40 mm, 2.90 mm, and 2.80 mm were recorded for 2%, 4%, 6%, and 8% replacements respectively. These conform to NGSRB limits of 2–4



mm for wearing courses. This suggests that fly ash incorporation can control deformation under loading, which is critical for long-term pavement durability.

Additionally, the relatively consistent flow range across different percentages indicates stable flexibility that can accommodate varying traffic loads. Such controlled flow is advantageous in reducing rutting potential, especially in tropical climates with high

temperature fluctuations. The results also imply that using fly ash as a partial replacement for conventional fillers could lower material costs while maintaining compliance with design standards.

This makes it a viable option for sustainable pavement construction, particularly in developing economies seeking cost-effective solutions.

Table 5: Asphalt Institute Criteria for the Marshall Mixture Design Method, 1997

Mix Specification	Heavy traffic (greater than 106 ESAL)		Light traffic (less than 104 ESAL)		Medium traffic (104-106 ESAL)	
	Min	Max	Min	Max	Min	Max
Stability (Minimum)	6672N	-	2224N	-	3336	-
Flow (0.25mm)	8	16	8	20	18	-
Air Void (%)	3	5	3	5	3	5
Compaction (50 blows each side)	35	50	-	-	75	-

Note: EASL is the Equivalent Single Axle Load.

Source: Asphalt Institute

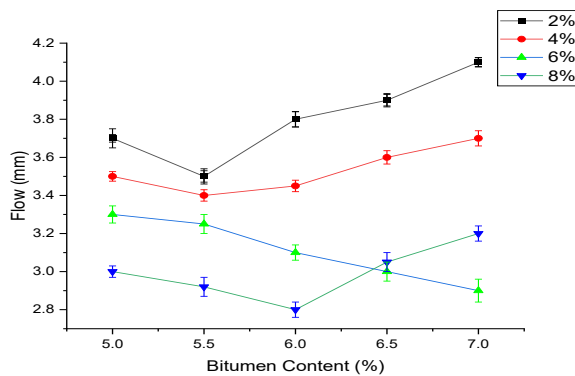


Figure 13: Marshal Flow of Fly ash Modified Asphalt against Bitumen Content

3.10 SEM/EDS Analysis of Steel Slag Modified Asphalt Concrete

The asphalt and steel slag mixture exhibits some separation of constituents, with tiny pits and cracks filled with asphalt to enhance embedding. Figure 13 reveals the porous structure of steel slag, which may influence the mechanical properties of asphalt mixtures due to increased binder consumption.

The primary elements identified were Ca, Fe, Si, and Mg, with calcium comprising over 60%, likely attributed to high flux dosages used in steel production [34]. This high calcium content can improve the

stiffness of the mixture, potentially enhancing resistance to permanent deformation under heavy traffic. However, the porosity may also lead to increased binder demand, affecting mix design economics and workability. These observations underline the importance of optimizing slag content to balance strength gains with cost and durability considerations.

3.11 SEM/EDS Analysis of Fly Ash Modified Asphalt Concrete

The asphalt and fly ash mixture exhibited a homogeneous phase with well-distributed fly ash, enhancing crack-mitigating behavior due to the spherical particles' incorporation into the bitumen matrix. Research indicates that fly ash-modified mixtures provide superior distribution of the asphalt membrane and hydration products. This dense structure results in low air void content, as illustrated in Figure 14 [45], [46], [47], [48]. The uniformity observed can contribute to better fatigue resistance and minimize moisture ingress, which is vital in wet climatic conditions. Furthermore, the presence of alumina and silica-rich particles suggests potential pozzolanic activity, which could enhance bonding at the interface over time. These microstructural benefits position fly ash as an effective sustainable additive for high-performance asphalt mixtures



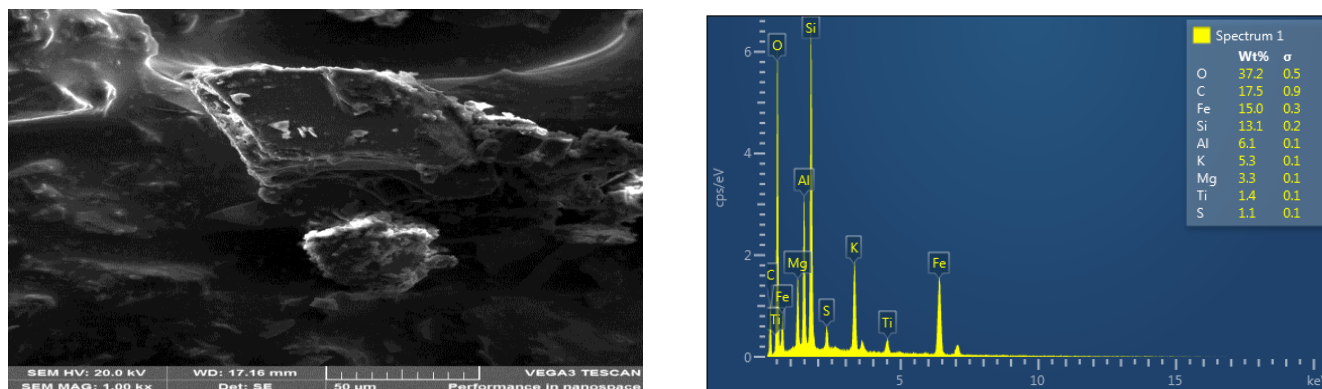


Figure 14: SEM/EDS Image of Steel Slag Modified Asphalt

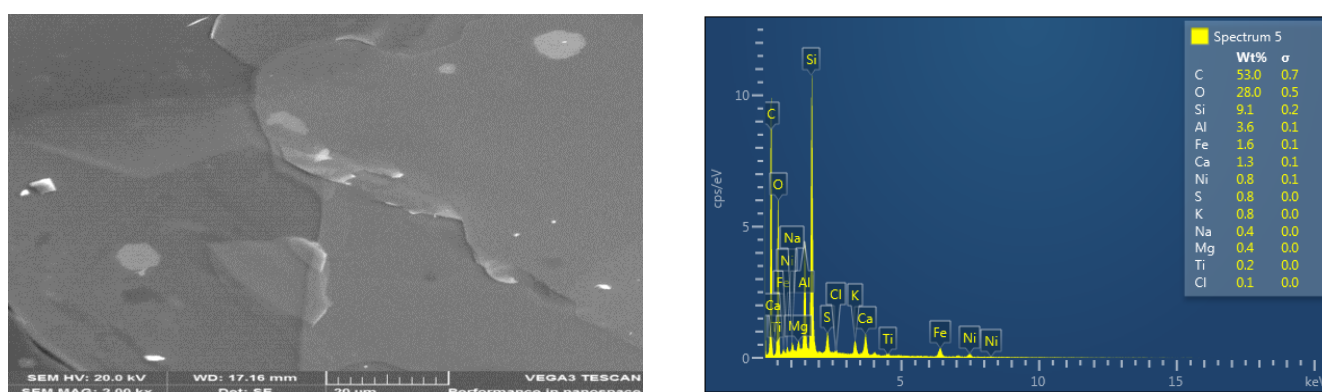


Figure 15: SEM/EDS of Fly Ash Modified Asphalt

4.0 CONCLUSION

This study investigated the performance of bituminous concrete mixes incorporating steel slag and fly ash from selected industrial sources. The materials were evaluated through physical, chemical, and microstructural characterizations, alongside Marshall stability and flow tests.

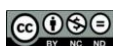
Key findings include:

- The physical properties of the wastes met Federal Ministry of Works and Highway Manual (2013) specifications, while bitumen rheological properties adhered to ASTM standards.
- XRD confirmed crystalline–amorphous phases, while TGA showed thermal stability up to 820°C, indicating suitability for high-temperature pavement conditions.
- Marshall stability peaked at 13.00 kN for steel slag-modified asphalt (8% slag at 6% bitumen) and 10.6 kN for fly ash-modified asphalt (8% fly ash), exceeding the 3.5 kN NGSRB minimum requirement.

- Flow values ranged from 2.8–3.5 mm for both modifiers, aligning with NGSRB's acceptable limits of 2–4 mm for wearing courses.
- SEM/EDS revealed that fly ash formed a dense, uniform matrix reducing voids, while steel slag showed porosity and micro-cracks that could enhance binder interaction but may require controlled use.

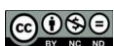
These results suggest that, within optimal replacement levels, steel slag and fly ash can be adopted in wearing courses and binder courses for medium- to heavy-traffic roads. Their stability and flow characteristics indicate resistance to rutting and cracking, while the thermal resilience supports use in regions with fluctuating temperatures.

However, these conclusions are specific to the quarry wastes studied; properties of steel slag and fly ash differ significantly across sources due to variations in production processes. As such, these results cannot be generalized without further characterization of alternative sources. Broader research is recommended to validate field performance and economic feasibility across diverse quarry waste streams.



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