# Static Mechanical Properties of Bituminous Concrete Modified with Tire Powder, Cross-Linked Plastic Bottles and Sulphur

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**Abstract:** ***This study examines the impact of tire powder and cross-linked plastic bottles combined with sulphur on the static mechanical properties of bituminous concrete. Traditionally composed of aggregates and bitumen, bituminous concretes are widely used for asphalt pavements. However, their degradation often results from the combined effects of climatic conditions, traffic loads, as the inherent limitations of the material used. Despite advancements such as the development of Enhanced Modulus asphalt (EMA) and the incorporation of additives like plastic, rubber, and sulphur to enhance pavement performance, these issues persist. To further improve the mechanical properties of bituminous concrete, this study explores the use of cross-linked powders derived from plastic bottles and tires. To evaluate the variations in the static mechanical characteristics of the modified materials, the components were characterised according to relevant standards. Bituminous mixtures were then manufactured and subjected to Duriez and Marshall tests. The modified composites were obtained by replacing 5%, 10% and 15% of the bitumen with a blend of tire powder, plastic bottle powder and sulphur in the following proportions of 40%, 28% and 32% respectively. The results indicated a decrease in the penetrability of neo-binders with the incorporation of 15% modified class 35/50 bitumen. Additionally, the simple compressive strengths of the modified composites were as follows; 9.81 MPa for the $BB_5$, 9.93 MPa for the $BB_{10}$ and 9.99 for the $BB_{15}$, all exceeding the compressive strength of the control asphalt concrete ($BB_0$), which is 9.75MPa. Moreover, while $BB_0$ has a stability of 961 kg, each of the modified composites demonstrated greater stability, notably $BB_5$, $BB_{10}$and $BB_{15}$which have respectively for stability at $975\ kg$ ; $989,9\ kg\ et\ 999,99\ kg$. Thus, bituminous neo-concrete has the potential to be used in the construction of more durable pavements.***

**Keywords:** Bituminous concrete, tires, plastic bottles, cross-linking

## 1. Introduction

The degradation of asphalt pavements is often linked to the deterioration of their mechanical properties. This degradation manifests in various forms, including material tearing, cracking, and irreversible deformations [1, 2]. Improvements in the quality of bituminous composites have led to the development of new manufacturing techniques and processes, resulting in products such as Enhanced Modulus Asphalt (EMA) [3], bituminous concrete enriched with plastics [4, 5, 6, 7, 8, 9, 10,11], rubber, and sulphur [11, 12, 13, 14, 15, 16, 17]. Despite these advancements, resistance to mechanical stresses remains a challenge, as evidenced by the persistence of the aforementioned pathologies.

Cross-linking with sulphur [16, 18, 19] has been shown to significantly enhance certain mechanical properties of plastics [16, 20, 21] and rubbers [22, 23], both of which are abundant in waste materials. This approach not only improves material performance but also contributes to environmental sustainability through the use of waste tires [24] and plastic bottles [15, 20, 24]. When used individually in bituminous concrete, sulphur, plastic and rubber [24, 25] generate composites that leverage their respective advantages, which warrant further enhancement.

This study evaluates the influence of the partial substitution of bitumen in BBSG 0/10 by 5%, 10%, and 15%, using a mass ratio of tire powder, plastic bottle powder, and sulphur at 40%, 28%, and 32%, respectively, through the Duriez and Marshall tests.

## 2. Materials and Methods

### 2.1. Materials

The components used in this study include aggregates, bitumen, and partial bitumen substituents (sulphur, tire powder and plastic bottle powder). The basic bitumen is produced by TOTAL according to the NF EN 12 591 [26] standard, which specifies that the penetrability, softening point, and mass variation fall within the ranges of [50; 70], [46; 54], and ]0; 0,5], respectively. The granite aggregates are sourced from the AKAK ESSE quarry and are used for asphalting the NR 17A Mengong – Sangmelima in the South Cameroon region.

The tire powder (fig 1) is obtained by screening through a 1 mm sieve, and is subjected to magnetic purification. This powder is derived from the shredding of non-reusable used tires (PUNR), which have been previously transformed into small pieces, cleaned, and dried.


**Figure 1:** Obtaining tire powders

The powder from plastic bottles, specifically polyethylene terephthalate (PET), is produced by transforming these bottles - non-opaque, transparent and coloured - into flakes. This process involves thoroughly washing the bottles with water, followed by wringing and drying that (fig 2 (a), (b), (c)). The dried flakes are then melted, cooled, and ground (fig 2: (d), (e)) and sift through a 1 mm mesh sieve.


**Figure 2:** Transformation of PET waste into powder

The sulphur used in this study was extracted using the fresh process and subsequently reduced to fines to facilitate accurate weighing. This extraction method minimises impurities by introducing three (03) pipes into the sulphur deposit. Very hot steam is injected through one of the outer pipes, while cold water is introduced through the other pipe. This process causes the sulphur to rise through the central channel.

### 2.2. Methods

The study aims to characterise the conventional components and the control bituminous concrete after its manufacture, which precedes the formulation. Additionally, it includes the determination of the characteristics of modified bituminous concretes derived from neo-binders.

#### 2.2.1. Control Bituminous Concrete

The control bituminous concrete is composed of aggregates and bitumen that conform to current standards. Its Duriez and Marshall parameters will be compared with those of the modified bituminous concrete.

##### *2.2.1.1. Component Characterisation*

It consists in determining the following parameters according to the relevant standards:

**1) Water Content**

The aim is to determine the water content of the aggregates by drying them in a ventilated oven, in accordance with the NF P 94-050 [27] standard. The procedure involves taking washed material and heating it at (110 ± 5) °C until a constant mass is achieved, with a permissible difference of 0.1%.



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**2) Particle Size Analysis**

Conducted according to the NF P 94-056 [28] standard, particle size analysis involves separating the components of a material based on their size using a series of sieves with square meshes. These sieves are arranged in ascending order of meshes size, from bottom to top. *The material is first washed to remove all fines ( $< 80\mu m$),* then dried in an oven for 24 hours. The purpose of the test is to represent the size and the respective percentages of the different grain families constituting the material sample through a particle size curve. This curve illustrates the percentages of the material retained on each sieve or the refusals based on the mesh sizes.

For particles smaller than 80 microns, sedimentometry is employed for particle size analysis. The results allow the determination of the percentages of each aggregate in the mixture, facilitating the generation of a granular spindle to optimize formulation.

Two parameters derived from the particle size curve are the coefficient of curvature (Cc) and the coefficient of uniformity (Cu), which provide insights into the distribution of granular diameters.

Two (02) parameters are determined of the particle size curve These are the coefficient of curvature ($C_c$) and the coefficient of uniformity($C_u$) which will inform on the display of granular diameters.

**3) Los Angeles Coefficient**

The Los Angeles coefficient is determined according to the NF P 18-573 [29] standard. This coefficient assesses the resistance of aggregate samples to fragmentation caused by impact. The test involves subjecting the material to standardised ball shocks in a Los Angeles machine for 30 minutes, after which the amount of material passing through a 1.6 mm sieve is measured. Prior to testing, the aggregates are washed and dried in an oven at 105 °C to eliminate all moisture.

**4) Micro Deval Coefficient**

Determined in accordance with NF P 18-572 [30] standard, the Micro Deval coefficient informs about the resistance of aggregates to wear. This is achieved by subjecting the aggregates to reciprocal friction with spherical balls ($10 \pm 0{,}5\ mm$ in diameter) within a rotating cylinder. Prior to testing, the aggregates are washed and dried in an oven at 105 °C. A mixture of 2.5 liters of water, along with the aggregates and abrasive charge—based on the granular class—is kneaded for 2 hours. After the test, the assembly is washed over a 1.6 mm sieve, and the refuse (without loads) is dried in the oven at 105 °C until a constant mass is reached.

**5) Flattening Coefficient**

The flattening coefficient is determined for aggregates ranging in size from 4 to 50 mm, in accordance with the NF P 18561 [31] standard. This coefficient characterises the shape of the aggregates. The test involves a two-step sieving process: first, using sieves with square meshes to classify the sample into different classes d/D0 specifying the mass of each class that is noted $M_g$. Then the second which is the sieving of the different granular classes d/D on grids with parallel slots of gauge E.

**6) Sand Equivalent**

The sand equivalent (ES) is evaluated on the fraction of aggregate passing through a 2 mm square mesh sieve, according to the NF P 18-598 [32] standard. This test provides information on the cleanliness of the sand. A dry mass of the sample (120 ± 1g) is introduced into a graduated cylindrical specimen containing a washing solution.

The mixture is stirred for 90 cycles (± 1 cycle) for 30 seconds (± 1 second). Subsequently, an additional amount of washing solution is added to clean the edges of the specimen and to cause the flocculate (dirt on the sand) to be suspended above the sedimented sand. After 20 minutes of sedimentation, three distinct layers are observed: sedimented sand, flocculate, and clean washing solution. The height of the sedimented sand ($h_2'$) with flocculate can be measured on the graduated specimen specimen. ($h_1$). Finally, a piston is lowered into the specimen until it rests on the sedimented sand, and the height of this sedimented sand is recorded as ($h_2$), also read from the graduated specimen.

***2.2.1.2. Formulation and Manufacture of control Bituminous Concrete***

The formulation, which determines the percentage of each granular class is carried out in accordance with the standard NF P 98-150-1 [33]. This process includes calculating the percentage of bitumen($P_{bi}$) calculated using the equation proposed by LALDJI in 2013.

$$P_{bi} = (TG + 120)/100$$

Where, *TG* called total particle size, is the sum of the percentages of passers-by from granular mixture to meshes 16, 12.5, 10, 6.3, 4, 2 and 0.080.

The manufacture of bituminous concrete is done in accordance with the standard NF EN 13 108-1 [34]. This phase consists of:

a) Mixing aggregates of different classes;
b) Kneading to ensure better dispersion of aggregates;
c) Heating the granular mixture;
d) Pouring the liquefied bitumen over the granular mixture, homogenising and proceeding to cure according to the class of bitumen used.

***2.2.1.3. Characterisation of Control Bituminous Concrete***

The characterisation is done in relation to the Duriez (NF P 98 251-1) [35] and Marshall (NF P 98 251-2) [36] tests.

**2.2.2. Modified Bituminous Concrete**

These composites, which are subjected to the same tests as the control bituminous concrete, are produced using a wet process. This process consists in manufacturing the bituminous concretes with neo-binders that are obtained after the formulation of the modified bituminous concretes.

***2.2.2.1. Formulation of Modified Bituminous Concretes***

Modified bituminous concretes are obtained by maintaining the same granular skeleton while partially replacing 5%, 10% and 15% of the bitumen with a mass sum of PUNR powder, PET powder and sulphur. This mass sum is composed of 40% PUNR powder, 28% PET powder and 32% sulphur.

#### *2.2.2.2. Manufacture and Characterisation of Modified Bitumen*

The modified bitumen is manufactured in chronological order of the four (04) steps below and according to standard NF EN 12 697-35 [37]:

a) Liquefaction of pure bitumen;
b) Addition of all partial substituents in pure liquefied bitumen;
c) Mixing for ten (10) minutes;
d) Spreading the mixture in a covered bowl for ten (10) minutes.

With regard to the characterisation of modified binders, only the penetrability test is carried out in relation to NF EN 1426: 2007 [38] standard, along with the classification of each modified binder.

#### *2.2.2.3. Characterisation of Modified Bitumen*

This involves determining the penetrability in accordance with the standard NF EN 1426: 2007 [38] and deducing the class of each modified bitumen.

#### *2.2.2.4. Manufacture of Modified Bituminous Concretes*

The manufacturing process for modified bituminous concrete includes the following steps:

a) Mixing aggregates of different classes;
b) Mixing to ensure better dispersion of the aggregates;
c) Heating the granular mixture
d) Introducing liquefied bitumen into the granular mixture, followed by homogenisation and curing according to the class of bitumen used.

#### *2.2.2.5. Characterisation of Modified Bituminous Concrete*

The static mechanical characterisation of modified bituminous concrete is conducted using the Duriez (NF P 98 251-1) [35] and Marshall (NF P 98 251-2) [36] tests.

## 3. Results and Interpretation

The results to be interpreted are the characteristics resulting from the tests conducted on the components (modified aggregates and bitumen) as well as on the bituminous concretes (both pure and modified) previously formulated and manufactured.

### 3.1. Aggregates

The particle size analysis (Table 1) shows the composition of the control mixture

**Table 1:** Particle Size Analysis

| Mesh Diameters | Granular Classes and Rates | | | Granular Mixture | BBSG0/10LCPC Granular Spindle | | Average Values |
|---|---|---|---|---|---|---|---|
| | 6/10 | 4/6 | 0/4 | | Min. | Max. | |
| 16 | 100 | 100 | 100 | 100 | 100 | 100 | 100 |
| 14 | 100 | 100 | 100 | 100 | 100 | 100 | 100 |
| 10 | 93.5 | 97.2 | 100 | 99.13 | 95 | 100 | 97.5 |
| 6.3 | 33.5 | 69.1 | 97.1 | 67.9 | 62 | 74 | 68 |
| 4 | 9.1 | 46.1 | 89.2 | 53.4 | 48 | 58 | 53 |
| 2 | 6.8 | 26.1 | 68.7 | 38.9 | 30 | 45 | 37.5 |
| 1.25 | 6.6 | 8.9 | 41.2 | 21.2 | 20 | 28 | 24 |
| 0.315 | 3.8 | 6.4 | 27.1 | 16.55 | 10 | 19 | 14.5 |
| 0.2 | 1.3 | 3.3 | 21 | 9.2 | 8 | 15 | 11.5 |
| 0.08 | 1.1 | 3.2 | 13 | 7.5 | 5 | 9 | 7 |

Data analysis presented in Table 1 indicates that the curve resulting from the values of the granular mixture is appropriately inscribed in the LCPC granular spindle for BBSG 0/10. This suggests that the aggregates are suitable for the manufacture of BBSG 0/10.

The coefficient of uniformity $(C_u = 23{,}70)$ and the coefficient of curvature $(C_c = 2{,}45)$ of the granular mixture meet the respective criteria $1 < C_c < 3$ and $C_u > 4$. This highlights a favourable distribution of granular diameters in accordance with the standard NF P 18–540.

Additionally, these aggregates have undergone a series of tests to determine essential properties (Table 2).

**Table 2:** Essential Properties of Aggregates

| | 0/4 | 4/6 | 6/10 | Specifications |
|---|---|---|---|---|
| Moisture Content W (%) | 3.60 | 2.81 | 1.89 | |
| CA (%) | ≠ | 14.8 | 15.2 | < 20% |
| LA | ≠ | 32% | 34% | < 35% |
| MDE | ≠ | 16% | 19% | < 25% |
| Sand Equivalent ES (%) | 80% | ≠ | ≠ | > 40% |

In accordance with the relevant standards, the properties listed in Table 2 confirm the suitability of these aggregates in the manufacture of quality bituminous concrete.

### 3.2. Modified Bitumen

Table 3 shows the penetrability of the modified bitumen and their respective classes.

**Table 3:** Properties of Bituminous Binders

| | $B_5$ | $B_{10}$ | $B_{15}$ |
|---|---|---|---|
| Penetrability at 25°C | 63.2 | 51.7 | 40.3 |
| Class | 50/70 | 50/70 | 35/50 |

It has been observed that the partial substitution of bitumen with the mass sum of PET powder, PUNR powder and sulphur, reduces penetrability to the point of causing a change in the class of the bituminous binder $B_{15}$ . This densification results from the interaction between the partial substituents and the bitumen, embellished by sulfuric cross-linking [13; 17] which, according to the work of Charles Goodyear in 1939, leads to a spatial entanglement of linear polymer molecules, increasing atomicity. This is preceded by the destruction of several multiple bonds and the disappearance of some benzene nuclei [13, 17].



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### 3.3. Formula of Bituminous Concretes

Noting $BB_i$ as bituminous concrete with i% bitumen substitution, the masses of the components per 10,000 g of bituminous concrete are given in Table 4.

**Table 4:** Component Masses per 10,000 g of BB

| | Aggregates | | | Binder Components | | | |
|---|---|---|---|---|---|---|---|
| | 6/10 | 4/6 | 0/4 | Bitumen | Plastics | Tyres | Sulphur |
| $BB_0$ | 3300 | 1420 | 4720 | 560 | 0 | 0 | 0 |
| $BB_5$ | 3300 | 1420 | 4720 | 532 | 8.4 | 63 | 12.6 |
| $BB_{10}$ | 3300 | 1420 | 4720 | 504 | 16.8 | 73 | 25.2 |
| $BB_{15}$ | 3300 | 1420 | 4720 | 476 | 25.2 | 84 | 37.8 |

### 3.4. Characteristics of Bituminous Concrete

This section presents a set of seven (07) parameters, as detailed in Table 5. Among these, four parameters are derived from the Duriez test (simple compressive strength, compactness, degradation coefficient and imbibition rate), while three parameters are obtained from the Marshall test (stability, creep and compactness).

**Table 5:** Duriez and Marshall Settings

| | Parameters | Samples | | | | Requirements |
|---|---|---|---|---|---|---|
| | | $BB_0$ | $BB_5$ | $BB_{10}$ | $BB_{15}$ | |
| **Duriez Settings** | $R_C$ *air (Mpa)* | 9.75 | 9.81 | 9.93 | 9.99 | $7 \leq R_{air} \leq 11$ |
| | $R'_C/R_C$ *coef. Dégradation* | 0.61 | 0.71 | 0.79 | 0.80 | $0.6 \leq R_{eau}/R_{air} \leq 0.85$ |
| | Average Compactness (%) | 93.34 | 95.40 | 96.32 | 97.3 | 90 – 95 |
| | Mean Imbibition (%) | 1.68 | 1.503 | 1.133 | 1.627 | ≤ 3% |
| | | | | | | |
| **Marshall Settings** | Average Stabilities (kg) | 961 | 975 | 989.9 | 999.9 | 800 à 1200 |
| | Creep Means | 3.5 | 3:01 | 2.92 | 2.86 | ≤ 4 |
| | Average Compactness (%) | 94.03 | 94.97 | 95.21 | 95.98 | 91 – 95 |

#### 3.4.1. Interpretations

The influence of partial bitumen substituents on parameters relating to the two tests carried out leads to a classification of these parameters into two groups. The first group includes imbibition and compactness (Marshall and Duriez), while the second group consists of stability, strength, coefficient of degradation and creep.

An analysis of Table 5 shows that:
1) Compactness increases with bitumen substitution rate, exceeding the upper limit bound;
2) Imbibition rate is almost inversely related to the percentage of bitumen substitution;
3) Strength of bituminous concrete in simple compression increases with the bitumen substitution rate;
4) The coefficient of degradation of bituminous neo-concrete is significantly boosted
5) Creep decreases relatively to the bitumen substitution rate;
6) Stability evolves in line with the bitumen substitution rate while remaining within the permissible interval.

The increasing evolution of the compactness of modified composites and the decrease in their imbibition rate highlight:
- The formation of simple bonds to the detriment of multiples, with the development of three-dimensional sulfide bridges (C–S or S–S) between linear polymers resulting from the destruction of multiple bonds as explained by the work carried out by Charles Goodyear in 1939;
- The impact of the wet manufacturing process, which, facilitates the effective digestion of the cross-linking phenomenon that begins during the manufacture of neo-binders which was done at 150°C, temperature around which the PET softens and the PUNR powder which better absorbs the heavy oils of the liquefied bitumen;
- A reduction of the dominant vacuolar structure in the bitumen-PUNR vortex by the softened PET powder close to this temperature (150°C)
- The considerable reduction in the vacuum rate by cross-linking due to the cementing power of the crosslinker which is sulphur;
- The hydrophobic power of the crosslinker;
- The drastic decrease in the honeycomb structure of modified bituminous concrete following cross-linking which is responsible for the densification of modified binders;
- The disappearance of benzene nuclei and the transition from multiple bonds to single bonds [9]. These mechanisms are accompanied by isotropic molecular aliasing resulting from the formation of bridge -S-S-, -C-C- and C-S true manifestation of cross-linking as informed by the work carried out by Charles Goodyear in 1939;
- A sharp increase in atomicity due to the establishment of a highly entangled molecular network [14], as a result of the interactions between the partial substituents and bitumen that begin during the manufacture of the modified binder.

## 4. Conclusion

This study highlights that the rolling layer made of bituminous concrete is consistently subjected to environmental stresses and traffic-related degradation. In response to these degradations, new technologies have been proposed to strengthen the characteristics of the bituminous composite, however, the pathologies reflecting its mechanical limits remain perceptible and persistent. As a result, the availability of non-reusable user tire waste and plastic bottle waste combined with cross-linking, motivated the evaluation of the impact of tyre waste and cross-linked plastic bottles on bituminous concrete. Modified bituminous concrete obtained by wet process results from the partial substitution of bitumen with a mixture of tire powders, plastic bottles and sulphur, in a control bituminous concrete formulated and manufactured according to the relevant standards.

In the light of the results obtained from the laboratory tests carried out on modified bitumen (the penetrability test) and for both pure and modified bituminous concretes (Duriez and Marshall tests), the following conclusions can be drawn:

- The partial substitution of bitumen by a mixture of non-reusable user tire powder, plastic bottle waste powder (transparent and coloured) and sulphur increases the consistency with possible impact on the class of the neo-binder compared to the reference binder,
- Modified bituminous concretes have significantly better intrinsic static mechanical properties compared to witness bituminous concrete,

In addition, modified bituminous concrete obtained as a result of a partial substitution of bitumen for the mixture of tire powder, PET powder and sulphur has an impact on several areas. The use of non-biodegradable waste material is a significant gain for the environment and for society with the development of the HIMO approach. It should be noted that the integration of sulphur also makes it possible to recover this material, which is often considered as waste in mining Ultimately, the said substitution has a strong economic impact in the construction of pavements because, the bitumen that makes road asphalting work more expensive is partially replaced by waste that floods the city and gives rise to a composite with better properties then allowing to increase the life of the structure.

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