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Performance Analysis of Bio-Based Asphalt Mixtures Containing Lignin

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Abstract

Given the need to promote the circular economy and sustainability, one of the main current trends in road materials construction is to employ industrial residues and by-products deriving from renewable sources as extender, replacement or modifier of bitumen, obtaining the so-called "bio-binders". As regards, lignin can represent a potentially attractive solution, because it is the most abundant natural biopolymer, available in large quantity and characterized by certain chemical similarity with bitumen. In this context, this study focuses on the evaluation of two dense-grade asphalt mixtures for binder layer made with bio-binders containing two different lignins, as partial replacement of bitumen. A preliminary phase allowed to optimize lignin content (30% by bio-binder weight) based on empirical test (i.e. dynamic viscosity, penetration and softening point tests) with the aim of maximizing the bitumen replacement and at the same time obtaining two bio-binders having a consistency similar to a reference plain bitumen. In the second phase, two bio-based mixtures were produced by using the before-optimized bio-binders in different attempt contents. After mixing, specimens were produced by means of a gyratory compactor at set gyrations. Then, the two lignin-based asphalt mixtures were compared with the reference mixture in terms of workability, Indirect Tensile Strength (ITS) and water sensitivity. Despite the bio-based mixtures revealed a slightly penalized workability, overall results showed that they are characterized by fully comparable performances to the reference one, allowing a reduction of the effective bitumen content.

Keywords: Bio-binders; lignin; asphalt mixtures; circular economy; sustainability.

1. Introduction

Road construction and maintenance sector is responsible for GHG emissions in the atmosphere, and one of the reasons is due to the large use of asphalt mixture containing significant amount of bitumen, which is a non-renewable source deriving from crude oil distillation [1]. Nowadays, circular economy and sustainability are the two main principles to be pursued, pushing the paving sector to look for new renewable solutions

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[2][3]. The current challenge is to totally or partially replace bitumen with 100% alternative green sources deriving from by-products of industrial processes (e.g. wood and paper), animal waste (e.g. swine manure), vegetal oils (e.g. soybean oil, palm oil, vegetable oil), microalgae and more, entailing the production of the so-called bio-binders [4]. This could lead to a substantial decrease of bitumen in road pavements, at the same time promoting energy saving, environmental as well as global cost benefits.

In this context, the use of lignin can be a viable solution considering that it derives from the pulp and paper industry waste, and it is renewable, environmentally friendly, widely available being one of the most abundant bio-polymer on hearth and easily handling as well. Lignin has gained a considerable interest over time, because it presents several chemical similarities with bitumen, basically in terms of hydrocarbon composition [5]. Furthermore, it has been proven that lignin may create particular three-dimensional bonds with bitumen, which result in improving performances [6] and being rich in phenolic groups, it should give important antioxidant contribution [7]. Nevertheless, high material variability basically related to the production method adopted and to the raw material origin, makes the lignin a quite complex material which can result in a difficult understanding of the effective interaction with bitumen.

Several experimental investigations were found to focus on a partial replacement of bitumen with lignin, whose content mainly ranged around 10%. From those studies, the reduction of the penetration grade and increase in softening point [8] proved the hardening effect of the lignin on bitumen entailing an expected change of rheological properties. Additionally, promising results in terms of aging resistance confirm the antioxidant capacity of lignin, along with an improved bond strength. Better rutting resistance at high temperature and increased elastic components were also found for the system bitumen/lignin [9], even though fatigue and low temperature performances seemed to be penalised . However, only few researches have moved towards higher replacement rates, showing undesirable effects due to lignin crystallization or segregation when content of lignin exceed 40% [10]. Moreover, even less is known in literature about the application of lignin-based bio-binders in asphalt mixtures, but the few existent investigations seem to confirm the role of lignin similarly to what was found for asphalt binders [11]. *Table 1* sums up the main findings founded by the mentioned laboratory experiments.

Given this background, this paper aims at studying the feasibility of using bio-binders containing lignin as partial bitumen replacement to produce asphalt mixtures. To this end, a first phase of the investigation was devoted to maximising the bitumen replacement with the aim of obtaining bio-binders comparable with a plain reference bitumen. Physical testing such as penetration, ring & ball softening point and dynamic viscosity were performed on two plain bitumens characterised by different consistency and partially replaced with two lignin sources having different origin. In a second phase, bio-based asphalt mixtures were produced by using the optimized bio-binders and tested for their performance in terms of workability, indirect tensile strength (ITS) and water susceptibility. Finally, performance comparison was carried out between the asphalt mixture produced with the bio-binders and those with the reference bitumen.

Table 1: Main findings of the mentioned laboratory experiments on lignin

Lignin feedstock	*Lignin state*	*Lignin content (% by weight)*	*Benefits*	*Drawbacks*	*Reference*
Organic and lignosulfonate	powder	5-7%	Stiffening effect Improved aging resistance	Early thermal cracking	Asukar et al. 2016
Kraft and corn stalk	powder	5%	Stiffening effect, improved elastic recovery, moisture and aging resistance, high- and low-temperature performances	Penalised fatigue	Xu et al., 2021
Water waste from hardboard panels	liquid	5-10-20-40%	Improved moisture and thermal susceptibility, rutting at high and fatigue at low temperatures	Excessive foaming and crystallized particles at 40%	Pérez et al. 2019
Rice hulls, black liquor and kraft	powder	2-4-6%	Improved high temperature PG, long-term aging index, rutting resistance	Penalized fracture energy	Arafat et al. 2019

2. Experimental program

2.1 Binder phase

A conventional 50/70 pen grade bitumen usually employed at Italian latitudes was selected as the reference material, whereas two softer bitumens, having a 70/100 and 100/150 pen grade respectively, were adopted for generating bio-binders. *Table 2* summarizes the main physical properties of the three selected plain bitumens. Two different dark brown powder lignins of about 1.35 g/cm^3 density, deriving respectively from Sweden and Baltic countries (coded as S and B), were selected to produce the bio-binders *(Figure 1)*. In particular, the two softer bitumens were mixed with both lignins, at content levels of 10, 20 and 30% by bitumen weight with the aim of evaluating the lignin hardening effects on bitumen and producing two bio-binders having physical properties comparable to the reference bitumen. The preparation of bio-binders was performed according to a standardized mixing procedure as follows. Specifically, each bitumen sample was heated in a forced-draft oven for one hour at a controlled equi-viscosity temperature previously determined from the dynamic viscosity testing of plain bitumens. Concurrently, lignin was finely crumbled and dried at 105°C to eliminate the moisture. Then, dried lignin was added gradually and dispersed through a high shear stirring mixer at a speed of 5000 rpm for about 30 minutes to obtain a homogenous bituminous blend. Afterward, all the bio-binders were tested in terms of penetration (EN 1426) and softening point (EN 1427) both in unaged and short-term aged conditions simulated through the Rolling Thin Film Oven Test (EN 12607-1). Moreover, viscosity of unaged binders was evaluated at 160°C through a Brookfield viscometer (EN 13302).

Finally, results were compared with Italian technical specification requirements for 50/70 bitumen [12], to identify the two most suitable bio-binders (derived from the different combinations of bitumens and type and content of lignin) for the laboratory production of the bio-based asphalt mixture.

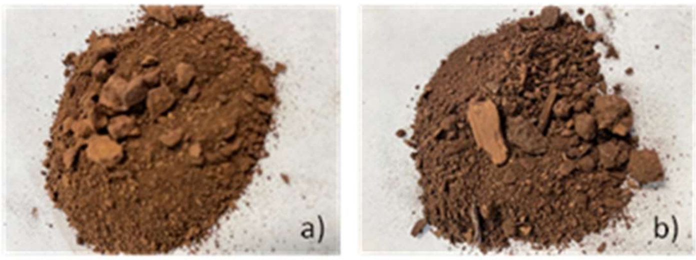

Figure 1: (a) lignin S; (b) lignin B.

Table 2: Characteristics of the plain bitumens evaluated during the binder study.

Bitumen	*Penetration (0.1 mm)*	*Softening point (°C)*	*Viscosity 160°C (mPa·s)*
50/70	52	51.5	116
70/100	88	48.0	117
100/150	127	45.9	88

2.2 Bio-based mixtures

In the second phase, the optimized bio-binders were used to produce two bio-based asphalt mixtures for binder course with a maximum aggregate size of 20 mm according to Italian Technical specifications [12]. Moreover, a reference mixture was produced by using the plain 50/70 pen grade bitumen for comparison purpose. Limestone aggregates for coarse, fine fractions and filler and 15% of Reclaimed Asphalt (RA) were employed and combined for obtaining the design granular blend *(Figure 2)*. RA consists of an unfractioned reclaimed blend having a bitumen content of 4.5%. For all the asphalt mixtures the same aggregate gradation was employed, whereas the binder contents were different (Table 3). Mixtures were produced in laboratory by means of a mechanical mixer by adopting production temperatures (mixing and compaction) deriving from equi-viscosity conditions in accordance with the NCHRP 648 specification [13]. After mixing, loose mixtures were aged in a forced-draft oven for 4h at 135°C to simulate the short-term aging (AASHTO R 30-02). Then, a gyratory compactor (GC) was used to produce cylindrical specimens with a diameter of 100 mm by setting a number of gyrations equal to 180 according to Italian Technical Specifications for dense-graded asphalt mixture [12] in order to obtain a set of specimens for a workability analysis. In particular, workability analysis, based on Compaction Energy Index (CEI) and residual air voids content assessment, allowed to select for each asphalt mixture a proper binder content resulting in a comparable compactability among mixtures. Then, a mechanical characterization was carried out on the asphalt mixtures produced with the "optimized" binder content determined in the previous phase and GC specimens were compacted at a residual air voids content of 6±0.5%. Such a set of specimens were subjected to ITS test in both "dry" and "wet" conditions, to evaluate the moisture sensitivity of the mixtures as well. Wet condition consisted in water conditioning specimens at a temperature of 40°C for 72 hours (EN 12697-12).

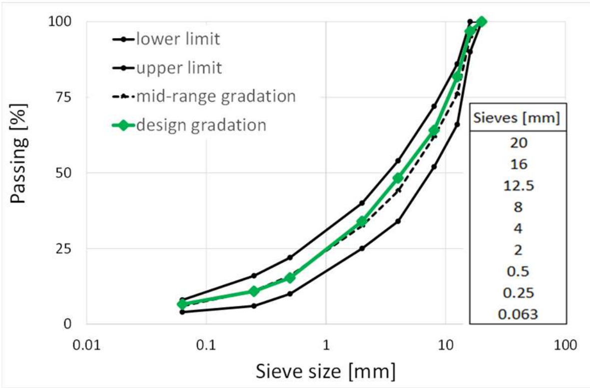

Figure 2: Design aggregate gradation for all the mixtures.

3. Test methods

3.1 Compactability and volumetric properties

The study of workability was performed by analyzing the Compaction Energy Index (CEI) and the residual air voids. For each investigated mixture, CEI can be calculated using the GC data recorded during laboratory compaction of specimens up to 180 gyrations and can provide an idea of the compaction efforts needed to achieve a certain target density [14]. Specifically, from the densification curve (*Figure 3*), CEI was determined as the area under the curve from the 8th gyration (considered to simulate the effort applied by the paver during laying down of mixture) to 92% maximum density ρ_{max} (simulating the completion of construction, when the pavement is open to traffic).

Mixtures with lower values of CEI show better workability, at the same time, too low values of CEI could be an indication of a tender mixture and should be avoided. Concurrently, the air voids content (V_m) was determined for each specimen based on its bulk density (EN 12697-06, Procedure A) and the maximum density (EN 12697-05, Procedure C) of the corresponding mixture.

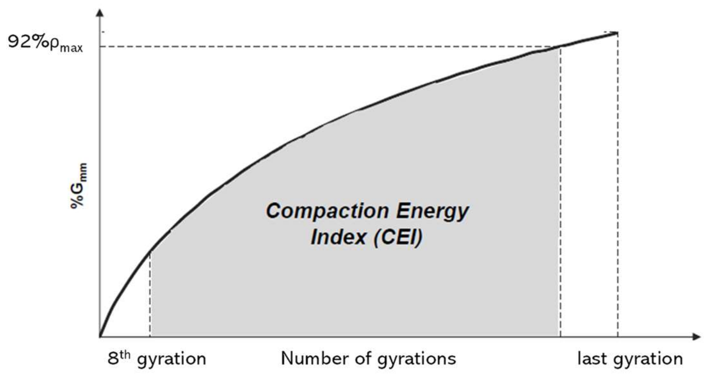

Figure 3: Densification curve to determine the Compaction Energy Index (CEI)

3.2 Indirect Tensile Strength

ITS tests were performed at 25 °C according to the EN 12697-23 standard. Specifically, GC cylindrical specimen was subjected to a compression force applied diametrically at a constant speed of 50 mm/min until it reached the failure. The ITS was calculated as follows:

$$ITS = \frac{2P}{\pi Dh} \quad [\text{MPa}] \tag{1}$$

where P is the peak load at failure in N, D and h are the specimen diameter and height in mm, respectively. Before testing, "dry" specimen series were conditioned in a climatic chamber for at least 4 h at 25 °C, whereas "wet" series were preliminary subjected to the conditioning in a water bath at 40°C for 72 hours. Four replicates for each testing condition were tested. Moreover, the water sensitivity was assessed by comparing the ITS values of dry and wet specimen series by calculating the Indirect Tensile Strength Ratio (ITSR), defined as:

$$ITSR = \frac{ITS_{wet}}{ITS_{dry}} \cdot 100 \tag{2}$$

where ITS_{wet} and ITS_{dry} refers to the average strength value of wet and dry samples, respectively.

4. Results and discussion

4.1 Binder phase

Different lignin contents were added with the main aim of producing bio-binders which were characterised by a consistency range typical of the reference bitumen (i.e. 50/70 pen. grade); the preparation was started from the two softer bitumens (70/100, 100/150), while maximizing the bitumen replacement content. As regards, Figure 4 shows the change in penetration value of the softer bitumens after being added by different percentages of lignin, along with consistency technical limits [12]. As can be noted, the lignin addition causes a hardening effect, and decrease in penetration is dependent on the type of bitumen and lignin. Although more than one bitumen-lignin combination met consistency requirements, the ones that maximised the amount of bitumen replacement (i.e. 30%) were the 70/100 bitumen combined with lignin S and 100/150 bitumen with lignin B.

Based on this experimental evidence, the two bio-binders, named as 70/100_S30 and 100/150_B30, were selected and subjected to further consistency tests (penetration and softening point) in unaged and short-term aged conditions, and viscosity tests in unaged conditions. Results are summarized in *Figure 5* along with the corresponding technical specification requirements for the reference 50/70 plain bitumen.

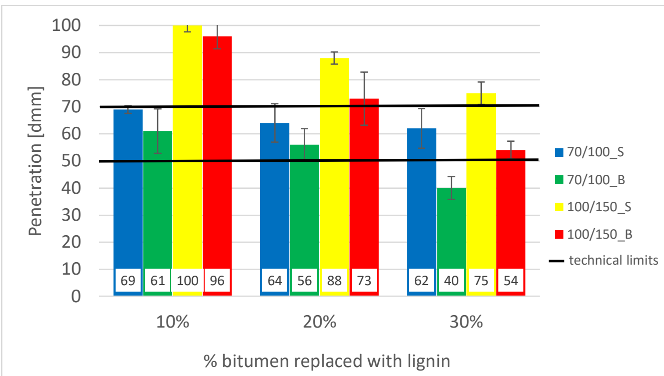

Figure 4: Penetration test on bio-binders containing different amount of lignin

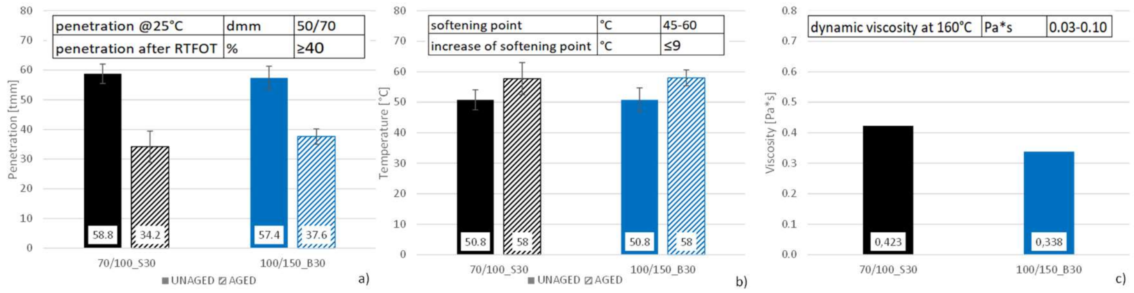

Figure 5: Consistency measurements: a) penetration test results, b) ring & ball test results and c) dynamic viscosity results, and technical specification requirements for 50/70 bitumen

From the analysis of results, it emerges that despite consistency requirements at intermediate and high temperature are satisfied, both bio-binders showed a viscosity 3-4 times higher than the allowed limits for 50/70 plain bitumen. The increased viscosity is as expected due to the presence of a solid phase (i.e. lignin) dispersed into the bitumen.

From a practical standpoint, this finding implies increasing mixing temperature to make the bio-binders more fluid and thus satisfy production needs. Based on the acceptance criteria by technical specification, 70/100_S30 and 100/150_B30 bio-binders could be considered equivalent to a plain 50/70 pen grade bitumen in terms of performance, therefore they have been selected to produce bio-based asphalt mixtures according to the experimental program.

4.2 Bio-based Asphalt mixtures

Bio-based asphalt mixtures and reference mixture were coded on the basis of the binder used, i.e. M70/100_S30, M100/150_B30 and M50/70, respectively. Several series of mixture were produced by using different binder contents, finally all the mixtures for testing characterization were optimised in terms of binder content on the basis of the compactability and volumetric analysis. According to technical specification for an asphalt mixture for binder layer [12] the total bitumen content should be in the range of 4.1-5.5% by aggregate weight. For this reason, the reference mixture M50/70 was produced and examined by using three trial total binder contents equal to 4.2, 4.8 and 5.4%. These contents also include 0.6% bitumen coming from the 15% RA fraction used within mixture. As for bio-based mixtures, it is right to remark that the total bitumen content consists of bitumen from RA and a bitumen content equal to 70% of the total added bio-binder amount. Thus, higher contents of total binder (i.e. bio-binder + bitumen from RA) up to 6.6% were selected for producing bio-based mixture in order to meet bitumen content requirements by technical specifications. *Table 3* summarizes the binder contents adopted for the reference and the two bio-based mixtures, by distinguishing among total binder of mixture, RA bitumen, fresh bitumen (i.e. added bitumen) and bio-binder content. Such a distinction allows a direct comparison between reference and bio-based mixture in terms of effective bitumen content within mixture.

Table 3: contents adopted to produce reference and bio-based mixtures.

Mixture	*Binder* [%]*	*RA bit. [%]*	*Fresh bit. [%]*	*Total bitumen [%]*
M50/70	4.2	0.6	3.6	4.2
	4.8		4.2	4.8
	5.4		4.8	5.4
M70/100_S30	4.8	0.6	2.9	3.5
	5.4		3.4	4.0
	6.4		4.0	4.6
	6.6		4.2	4.8
M100/150_B30	5.4	0.6	3.4	4.0
	6.4		4.0	4.6

**Binder: RA bitumen + Fresh bitumen + Lignin*

4.2.1 Air voids

Figure 6 illustrates air voids results obtained for all the mixtures as function of different binder contents.

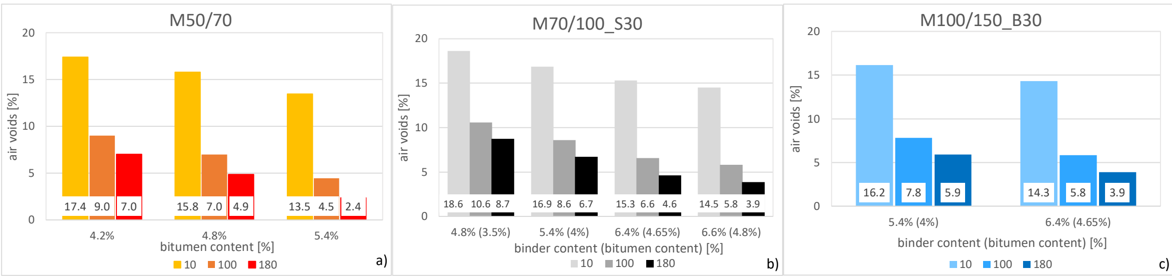

Figure 6: Air voids results for mixture: a) M50/70, b) M70/100_S30 and c) M100/150_B30

Specifically, air voids content at three number of gyrations (i.e. 10, 100 and 180) are reported according to the technical standard, which specifies the following requirements at each number of gyrations: 11-15%, 3-6% and ≥ 2%, respectively. It is evident that the higher the binder (i.e. bitumen) content, the lower the air voids percentage for all the mixture, thus confirming the lubricating role of bitumen in promoting the compaction of samples. As for the reference asphalt mixture, a bitumen content of 5.4% allows the specification ranges to be respected, whereas the same amount of binder for both bio-based mixtures result in too high residual air voids. This can be attributable to the significant lower content of available bitumen of bio-mixtures with respect to the reference one (i.e. 3.4% + 0.6% = 4% vs. 5.4%, see Table 3). The analysis of M70/100_S30 and M100/150_B30 mixtures produced at higher bio-binder contents, (i.e. 5.8 and 6.0% and only 5.8% respectively) thus containing a higher available bitumen content, confirms the prevalent role of bitumen for achieving an effective compaction, which could guarantee the compliance with technical requirements. However, M100/150_B30 mixture seems to show a slightly higher propensity to be compacted than M70/100_S30, this is likely due to its softer base bitumen rather than type of lignin used.

From the overall results, it can be stated that despite bio-based mixtures require a higher binder content to meet air voids requirement, the use of lignin-based binder would allow a saving up to 15% in terms of fresh bitumen as compared to the fresh bitumen content used in the reference asphalt mixture.

4.2.2 Workability

The Compaction Energy Index (CEI) was determined as representative parameter of the compactability of the reference and bio-based mixtures. Specifically, CEI values were depicted as function of different total bitumen contents tested for each asphalt mixture (*Figure 7*). For all the mixtures, as expected, the CEI values decrease as the bitumen content increases, highlighting the role of bitumen in promoting the workability of the mixtures. By comparing the CEI results among all the mixtures, it can be highlighted that the M70/100_S30 bio-based mixture shows a better compactability with respect to the reference mixture as the same total bitumen content is considered (i.e. 4.8%), pointing out that the presence of lignin does not hinder the compactability of the mixtures.

Whereas, from the comparison between the reference mixture produced with 5.4% of total bitumen and bio-based mixtures produced with a lower bitumen content, i.e. 4.6% and 4.8% respectively with the view of reducing the bitumen amount within the mixture, it emerges that the M50/70 mixture appears slightly more workable than the two bio-based mixtures and that M100/150_B30 seems to be slightly more workable than M70/100_S30, confirming what was found during the air voids study. Based on these experimental findings, it clearly emerges the primary role of the available bitumen to guarantee an effective compactability of mixture. However, both bio-based mixtures show a compactability aptitude which is comparable to the reference mixture, despite they contain lower contents of bitumen (4.6-4.8% vs. 5.4%).

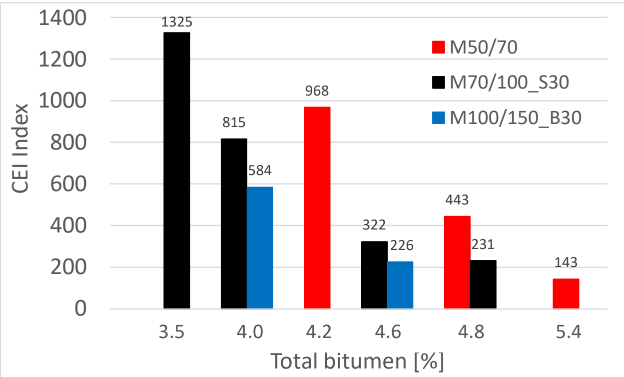

Figure 7: CEI results for all investigated mixtures.

4.2.3 Indirect Tensile Strength

Based on the volumetric and CEI results, a bitumen and binder contents respectively equal to 5.4% and 6.4% (4.6% of total bitumen content) were selected for the reference and two bio-mixtures, to produce further series of cylindrical specimens characterized by the same residual air voids content of 6±0.5% to be tested for strength and water sensitivity analysis. Figure 8 displays the ITS test results in dry and wet conditions for all the investigated asphalt mixtures. As far as both dry and wet conditioning are concerned, it can be observed that the mixtures produced with bio-binders showed slightly lower ITS as compared to the reference mixture especially for M100/150_B30, even though these differences are not significant. This comparable response between the reference and bio-based mixtures highlights how the lignin allows to compensate the potential reduced strength response of bio-mixtures deriving from the softer consistency of the base bitumen employed, consistently with the results obtained in the binder phase section. As regards wet condition, water conditioning does not seem to negatively affect the strength of all the mixtures, on the contrary a slightly improved ITS is observed for the bio-mixtures with respect to the dry conditions. This finding means that adhesion interactions between bitumen and aggregates are not penalized despite a replacement of bitumen by lignin. The observation is consistent with other research studies [15].

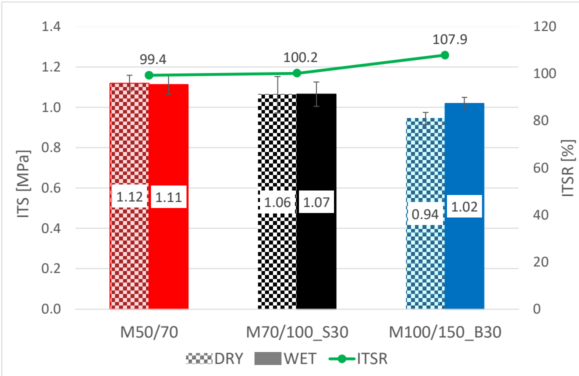

Figure 8: ITS results in dry and wet condition at 25°C.

5. Conclusion

The objective of this study focused on the feasibility of producing asphalt mixtures by using bio-binders obtained by maximizing the lignin content as partial bitumen replacement and starting from different bitumen and lignin sources. To this end, a first phase of the investigation was devoted to prepare bio-binders having a consistency comparable with a reference plain bitumen. In the second phase, two bio-based mixtures were produced by using the optimized bio-binders content and then compared with the reference mixture in terms of compactability (air voids and CEI analysis), ITS and water sensitivity. Based on the results gathered, the following conclusions can be drawn:

- the use of lignin led to an increase in the consistency (reduction in penetration and increase in softening point) of base bitumens.
- 30% (by weight) of lignin was used to obtain bio-binders characterized by a similar consistency of the reference 50/70 pen grade bitumen regardless of type of lignin; however, a higher lignin content caused operative issues for producing homogenous bio-binder sample.
- based on the results of compactability analysis performed on the bio-based mixtures, the introduction of lignin as bitumen replacement did not excessively penalize workability as well as volumetric properties of the mixtures, while allowing to save the bitumen content up to 15% as compared to the reference mixture.
- Strength response of asphalt mixtures was not negatively affected by the use of bio-binders, which results in comparable performance to that of the reference mixture even after water conditioning.
- The high water resistance of bio-based mixtures highlighted that the lignin did not affect the bond strength between bitumen and aggregate despite the lower effective bitumen content.

The positive findings, that will be followed by a more extensive performance-based characterization of bio-asphalt mixtures, represent the first step towards the use of lignin-based binders as a valid alternative to traditional bitumen to produce asphalt mixtures according to the basic principles of circular economy and sustainability.

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