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Comparison of the moisture damage of bituminous binder coupled with glass and limestone substrate using pull-off test

Éric Lachance-Tremblay, Michel Vaillancourt, Daniel Perraton, and Hervé Di Benedetto

Abstract: In this paper, the moisture susceptibility of different bituminous binders with two substrates (glass and limestone) was investigated. To that end, the tensile strength of different combinations of bituminous binder–substrate bond was measured using a pull-off test. This test was adapted from the pneumatic adhesion tensile testing instrument (PATTI) test to improve repeatability. Samples were tested in dry condition and after a 7-day conditioning in hot water bath (60 °C). An analysis of variance (ANOVA) was performed on the test results. Overall, the results show that in dry condition, the pull-off strength is a function of the bituminous binder type rather than of the substrate type. After water conditioning, an increase in the pull-off strength was observed for the bituminous binder without polymers and coupled with glass substrate. This was associated with an increase in binder stiffness. For the limestone substrate, the effect of water conditioning was significant only for one type of binder.

Key words: bituminous binder, glass, limestone, moisture damage, pull-off test.

Résumé : Dans le cadre de cette étude, on a examiné la vulnérabilité à l'humidité de différents liants bitumineux avec deux substrats (verre et calcaire). À cette fin, la résistance d'adhésion de différentes combinaisons de liant bitumineux–substrat a été mesurée au moyen d'un essai en arrachement. Cet essai est une adaptation de l'essai avec le « pneumatic adhesion tensile testing instrument » (PATTI) dans le but d'améliorer la répétabilité. Les échantillons ont été soumis à l'essai à l'état sec et après un conditionnement de 7 jours dans un bain d'eau chaude (60 °C). Une analyse de la variance (« ANOVA ») a été effectuée sur les résultats d'essais. Dans l'ensemble, les résultats révèlent qu'à l'état sec, la résistance à l'arrachement est fonction du type de liant bitumineux plutôt que du type de substrat. Après le conditionnement à l'eau, une augmentation de la résistance à l'arrachement a été constatée pour le liant bitumineux sans polymères et couplé avec le substrat de verre. On a associé cette augmentation à un accroissement de la rigidité du liant. Pour le substrat de calcaire, l'effet du conditionnement à l'eau était important pour la combinaison avec seulement un des types de liant.

Mots-clés : liant bitumineux, verre, calcaire, dommages par l'humidité, essai en arrachement.

1. Introduction

Durability is one of the most important properties when designing materials for road structures. Moisture damage is commonly recognized as one of the most severe distresses for pavement materials (Baldi-Sevilla et al. 2017; Chaturabong and Bahia 2018; Hamed 2017; Mehrara and Khodaii 2013; Porot et al. 2016). Moisture damage is defined as a degradation of material mechanical properties due to the presence of moisture in a liquid or vapor state (Caro et al. 2008). For bituminous materials, the result of moisture damage is expressed as a loss of the adhesive bond between bituminous binder and aggregate and (or) loss of cohesion of the mastic (Caro et al. 2008; Chaturabong and Bahia 2018; Hamed 2017).

Failure due to moisture damage in bituminous materials can be separated into three categories: (1) cohesive failure in the binder film, (2) adhesive failure at the binder–aggregate interface, and (3) cohesive failure of the aggregate (Pinzon 2004). Cohesive failure in the binder film occurs when water reacts chemically with the binder and reduces its cohesion (Kanitpong and Bahia 2003). In the case of adhesive failure, it occurs when water weakens the

bond between the binder and the aggregate surface. This causes a separation of the binder film from the aggregate (Bahia et al. 2012). In the literature, it is generally agreed that moisture damage is partly cohesive and partly adhesive (Bahia et al. 2012; Kanitpong and Bahia 2003). For asphalt mixture, the moisture damage intensity is influenced by the characteristics of the materials (binder and aggregates) and the quality of the binder–aggregate adhesion. Adhesion between the binder and aggregates is a very complex phenomenon that cannot be explained by one theory, but rather, by a combination of multiple theories, including the following (Caro et al. 2008; Hefer and Little 2005; Kanitpong and Bahia 2003):

- Mechanical theory: liquid asphalt binder penetrates the aggregate pores and irregularities which provide a mechanical interlock;
- Chemical reaction theory: adsorbed asphalt binder may chemically react with the aggregate surface components;
- Electrostatic theory: adhesion is the result of attraction of an electropositive and an electronegative surface;

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- Interfacial energy theory: molecules at the surface of a material usually have an excess of energy compared to those from the bulk of the material. Due to this excess of energy, when two materials come into contact, the adhesion is energetically favorable because the system tends to reduce the interfacial free energy.

The effect of water on bituminous materials is described by six mechanisms: (1) detachment or debonding, (2) displacement, (3) dispersion, (4) film rupture and (or) microcracks, (5) desorption, and (6) spontaneous emulsification (Caro et al. 2008). An extensive description of these mechanisms can be found in the literature (Caro et al. 2008; Kringos and Scarpas 2005; Little and Jones 2003). Moreover, physical solicitations such as stress caused by traffic loading and (or) temperature variation, as well as water entrance in the pavement structure, can potentially impact bituminous mechanical properties (Mehra and Khodaii 2013).

Multiple test methods are used to evaluate the moisture susceptibility of bituminous materials. The AASTHO T283 test is based on indirect tensile strength measurements on asphalt samples before and after water conditioning (AASHTO 2014). However, this test method has multiple disadvantages: it is time consuming, test results are highly variable due to saturation level differences, a visual examination of the stripping is subjective, and the conditioning procedure does not simulate repeated generations of pore pressure under loading (Liang 2008). Another commonly used test is the Hamburg wheel track test, which is very popular for its simplicity, but does not directly relate with pavement field performances, and is susceptible to misinterpreting moisture susceptibility (Buss et al. 2016). Moreover, those test methods measure bulk properties of the mixtures rather than any specific change in the adhesion and (or) cohesion properties of the binder–aggregate system (Canestrari et al. 2010). Traditional testing methods cannot be considered to be sufficient predictors of the cause of stripping. A better understanding of moisture susceptibility can be achieved if the tests are performed directly on the binder–aggregate system (Canestrari et al. 2010). To that end, a practical method to measure the adhesive and cohesive properties of a binder aggregate, the pneumatic adhesion tensile testing instrument (PATTI), was developed by Kanitpong and Bahia (2003) and modified to improve its repeatability (Canestrari et al. 2010; Kanitpong and Bahia 2003; Meng 2010; Youtcheff and Aurilio 1997). Recently, many authors have published papers evaluating bituminous material moisture susceptibility through binder–aggregate interface bond strength measurements (Apeagyei et al. 2014, 2015; Chaturabong and Bahia 2018; Figueroa et al. 2013; Zhang et al. 2017).

The work presented in this paper is part of a major research program looking at the use of glass aggregates in hot mix asphalt. The paper thus aims to evaluate the adhesion between different bituminous binders and aggregate types. To that end, a pull-off test was used to measure the bond strengths of different bituminous binder and aggregate combinations. A total of six bituminous binders and two substrates (limestone and glass) were studied. Pull-off tests were performed both in dry condition and after a 7-day hot water curing (60 °C).

2. Experimental

2.1. Tested materials

In this research, six binders and two substrates were used. The two substrates were glass and limestone plate. The limestone plate was sawn from boulder-size limestone aggregates and polished to ensure a smooth surface comparable to a glass plate. Six binders were chosen with different performance grades (PG), crude sources, and polymer contents (Table 1).

2.2. Pull-off strength test procedure

The pull-off test is often used to measure the adhesion strength between a binder and an aggregate substrate. Youtcheff and

Table 1. Performance grade (PG), crude source, and polymer content of the tested bituminous binder.

PG grade	Crude source	Polymer content (%)
PG58-28	Source 1	0
PG64-22	Source 1	0
PG64-22	Source 2	0
PG64-22	Source 3	0
PG70-28	Source 1	2.2
PG76-28	Source 1	3.5

Aurilio (1997) successfully used the PATTI to evaluate the binder–aggregate bond strength. This test was initially developed to measure the adhesion between a paint, coating or other adhesives with a rigid substrate (Canestrari et al. 2010). For the bituminous binder, a pull-stub is glued with bituminous binder to an aggregate substrate. Then, a pulling tensile force is applied through a pneumatic system until failure occurs. The pull-off strength (kPa) of a binder–aggregate system is expressed as the force required to peel the pull-stub from the substrate surface. This test has the advantage of allowing water conditioning of samples prior to testing, as well as the interpretation of the failure mode (adhesive failure or cohesive failure). However, its main disadvantage is that the applied stress rate cannot be controlled (Zhang et al. 2017). Moreover, the stress–strain relationship cannot be established.

In this project, the PATTI pull-off test method was adopted. To apply and measure the traction force, an automatic portable Elcometer pull-off adhesion device was used (Fig. 1). This device has a hydraulic pump that allows a continuous tensile force rate application.

Custom-made 50 mm diameter brass pull-stubs were used (Fig. 2). The 300 μm edge along the perimeter allows for control of the binder film thickness, which has been shown to be important for repeatability (Kanitpong and Bahia 2003). Furthermore, four cuts were made along the perimeter edge to allow excess binder to flow out of the stub. For each binder–substrate combination, one set of samples was prepared for each of the two testing conditions (in dry condition and after 7-day conditioning in hot water bath at 60 °C, or wet condition). The temperature, as well as the water level of the water bath, were monitored daily to ensure that samples were always covered by water at the target temperature. It is important to note that for the tests done in wet condition, the samples were put in a 20 °C water bath after the 7-day hot water curing for thermal conditioning.

2.3. Sample preparation

For each bituminous binder, a specific heating temperature was chosen to reach the same viscosity (0.17 Pa·s) (Table 2). To prepare the samples, the pull-off stub, the binder, and the substrate were heated at a specific heating temperature. Then, a small quantity of binder (approximately 0.9 g) was dropped onto the substrate. A scale with a precision of 0.001 g was used to measure the amount of binder used. Immediately following that, the pull-off stub was placed on the binder such as to prevent air from being trapped between the stub and the binder. Samples were then cooled at room temperature for 24 h.

3. Pull-off strength test results and analysis

For all pull-off tests performed in this project, a direct tensile force rate of 370 ± 20 kN/s was applied. Three replicate tests were performed in dry condition, and three replicate tests after water conditioning (wet condition) for each binder–aggregate combination.

Fig. 1. Pull-off test adhesion device used: automatic portable Elcometer adhesion tester. [Colour online.]



Fig. 2. Brass pull-stub (50 mm diameter) with 4 cuts (13 mm) along the perimeter edge and 0.3 mm edge.

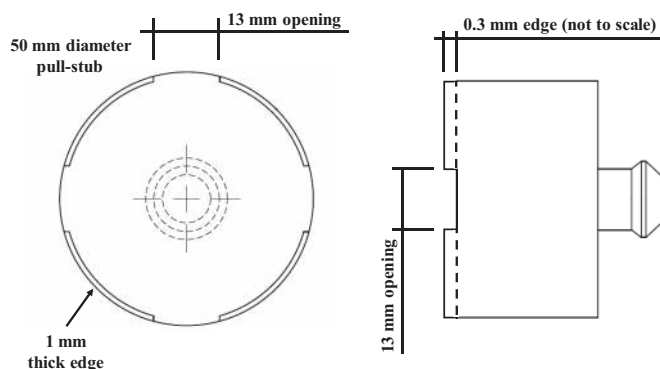


Table 2. Heating temperature used to prepare each binder–substrate sample.

Binder	Heating temperature for 170 mPa·s viscosity (°C)
PG58-28	151
PG64-22 # 1	160
PG64-22 # 2	160
PG64-22 # 3	158
PG70-28	170
PG76-28	170

3.1. Test results for dry condition

The average pull-off strength test results, as well as the coefficient of variation (CV %) in dry condition for all binder–substrate combinations, are presented in Table 3. Regarding the glass substrate, it can be seen that all three PG64-22 binders have higher pull-off strengths than the PG58-28 binder and the polymer modified binders (PG70-28 and PG76-28). In the case of the limestone substrate, a similar trend is observed: PG64-22 # 1 and PG64-22 # 2 have higher pull-off strengths than the other binders.

To try to explain why the three PG64-22 binders have different pull-off strengths, their respective complex shear modulus (G^*) was taken into consideration. Zhang et al. (2017) recently showed that in dry condition, the tensile strength of a binder–aggregate bond is dominated by the binder itself rather than by the aggregate type. Moreover, it was shown that the higher the G^* value,

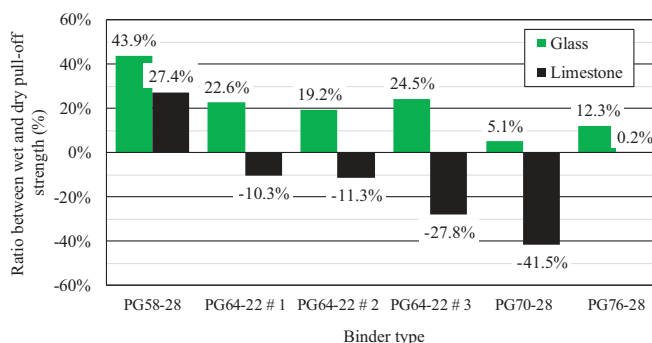
Table 3. Average pull-off strength and coefficient of variation for all binder–aggregate combinations tested in dry condition (three replicated per combination).

Binder type	Glass		Limestone	
	Avg. pull-off strength (kPa)	CV (%)	Avg. pull-off strength (kPa)	CV (%)
PG58-28	1145	9.0	1016	10.4
PG64-22 # 1	1907	10.0	1541	2.0
PG64-22 # 2	2198	6.8	2045	3.3
PG64-22 # 3	1614	4.7	1347	13.8
PG70-28	1476	6.2	1650	11.2
PG76-28	1291	7.0	1283	1.5

Table 4. Average pull-off strength and coefficient of variation for all binder–aggregate combinations tested in wet condition (three replicates per combination).

Binder type	Glass		Limestone	
	Avg. pull-off strength (kPa)	CV (%)	Avg. pull-off strength (kPa)	CV (%)
PG58-28	1648	3.0	1295	6.5
PG64-22 # 1	2337	9.1	1382	3.4
PG64-22 # 2	2621	2.0	1813	4.9
PG64-22 # 3	2009	5.8	972	1.0
PG70-28	1552	9.6	965	14.0
PG76-28	1450	3.1	1285	5.6

Fig. 3. Variation of the pull-off strength after wet conditioning for all binder–substrate combinations. [Colour online.]



the higher the pull-off strength. Regarding the three different PG64-22 binders in this project, their G^* at 64 °C are 1.30 kPa (PG64-22 # 1), 1.378 kPa (PG64-22 # 2), and 1.22 kPa (PG64-22 # 3), respectively. PG64-22 # 1 has the highest G^* and the highest pull-off strength. PG64-22 # 3 has the lowest G^* and the lowest pull-off strength, which is in agreement with what has been observed in other research studies.

3.2. Test results for wet condition

Each binder–aggregate combination was tested after water conditioning (7 days in a hot water bath at 60 °C). As a reminder, after water conditioning, samples were put in a water bath at 20 °C for thermal conditioning. Table 4 shows the average pull-off strength and the coefficients of variation (CV %) in wet condition. Significant differences can be seen when comparing the results of the two substrates; all pull-off strength values of the binder–limestone combinations are lower than those of the glass substrate.

A recent research study showed that the moisture damage of a given aggregate–binder bond is not solely affected by the substrate type (i.e., mineralogical composition), but also by the moisture content of the aggregate (Zhang et al. 2017). The authors mentioned that for moisture-susceptible aggregates, the moisture

Table 5. Failure surface pictures of all tested binder–substrate combinations in dry condition and after water conditioning.

Binder type	Glass		Limestone	
	Dry	Wet	Dry	Wet
PG58-28				
PG64-22 #1				
PG64-22 #2				
PG64-22 #3				
PG70-28			N/A	
PG76-28			N/A	

Note: N/A: Non-available due to data loss.

content is a relevant factor to take into consideration. The authors tested the pull-off strength of one binder, combined with a substrate, while varying the moisture content. Their results showed a strong correlation with the moisture content and the tensile strength reduction, and the higher the moisture content, the higher the decrease in pull-off strength. Another research study also showed that the degradation of the binder–aggregate bond is controlled in part by the amount of water that can diffuse from the aggregate into the binder–aggregate interface (Apeagyei et al. 2014). Apeagyei et al. (2015) identified the following characteristics for a susceptible aggregate–binder bond: high porosity, high moisture absorption, and high diffusion coefficient.

In the present research, the limestone absorption value is 0.5% and glass absorption is nil. In the case of limestone aggregate, water can reach the binder–aggregate interface not only through the sample boundaries, but can also diffuse through the limestone. In the case of glass, the water can reach the binder–aggregate interface only through the sample boundaries. Therefore, it can be assumed that a higher amount of water can reach the binder–limestone interface than in comparison with the glass aggregate. This could explain why limestone substrate pull-off strength values are lower than those of the glass substrate.

3.3. Variation of the pull-off strength after water conditioning

The variation of the pull-off strength after water conditioning was calculated with eq. (1) for all binder substrates (Fig. 3). A positive value means that there was an increase in the bond strength after water conditioning and a negative value means there was a decrease in the bond strength after water conditioning.

$$(1) \quad \left(\frac{\text{WET}_{\text{pull-off strength}} - \text{DRY}_{\text{pull-off strength}}}{\text{DRY}_{\text{pull-off strength}}} \right) \times 100$$

In Fig. 3, it can be seen that all binder–glass combinations have a positive value. This means that binder–glass bond strengths are higher after water conditioning. However, for polymer modified binders (PG70-28 and PG76-28), variation values are quite low. For the neat binders (PG58-28 and PG64-22), variation values range from 19.2% to 43.9%. For the limestone–binder combination, only the PG58-28 binder has a positive difference value (PG76-28 variation value of 0.2% is considered non-significant). For the other binders, the variation values range from –10.3% to –41.5%.

Fig. 4. Retained cohesive surface percentage of all binder–substrate combinations tested after water conditioning. [Colour online.]

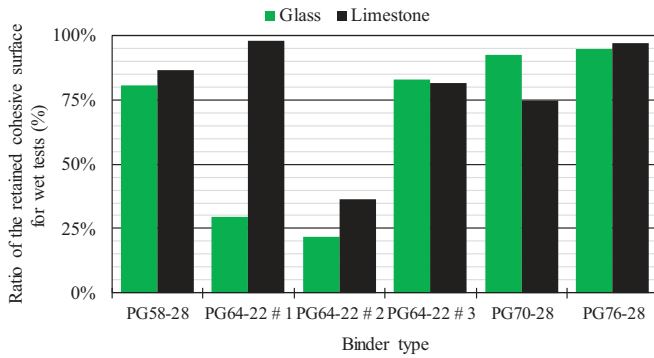


Fig. 5. Modification of the pull-off test device to include two LVDTs for displacement measurements. [Colour online.]



3.4. Effect of water conditioning on failure surface

In addition to the pull-off strength variation, the effect of water conditioning can be evaluated on the failure surface. The failure surface photographs of all binder–substrate combinations tested in dry and wet conditions are presented in Table 5. The failure surface can be interpreted either as the results of a cohesive failure, of an adhesive failure, or of a combination of cohesive-adhesive failures. In Table 5, it is clear that all binder–substrate combinations tested in dry condition show a cohesive failure. PG70-28 and PG76-28 binders with limestone pictures are not presented because of a computer data loss. Regarding the test done in wet condition, all failure surfaces show a combination of cohesive-adhesive failures.

Fig. 6. Relation between displacement and time during pull-off test: identification of the two different evolutions at phase 1 and phase 2. [Colour online.]

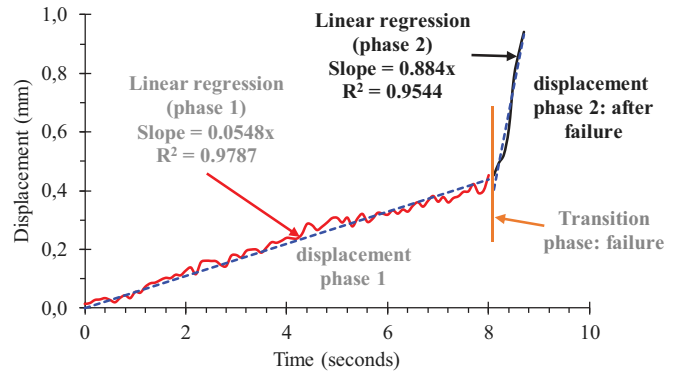


Fig. 7. Stress–strain relation for PG58-28 binder tested with glass substrate in DRY and WET conditions. [Colour online.]

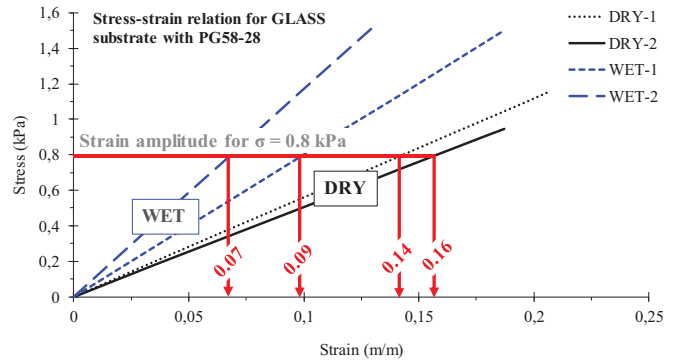
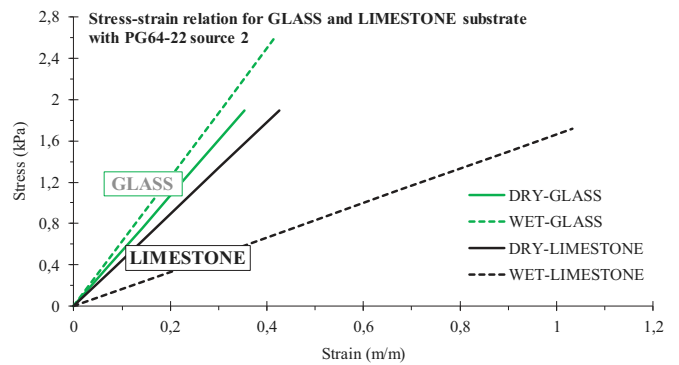


Fig. 8. Stress–strain relation for PG64-22 source 2 binder tested with glass and limestone substrate in DRY and WET condition. [Colour online.]



As proposed by Zhang et al. (2017), the retained cohesive surface percentages were calculated using a gray analysis in an image software application. This allows the quantification of the level of cohesive and adhesive failure mode (Zhang et al. 2017). Figure 4 shows the calculated retained cohesive surface percentage for all binder–substrate combinations tested in wet condition. For the glass substrate, the retained percentage values for polymer modified binder (PG-70-28 and PG76-28) are the highest (over 90%). This means that the surface failure in wet condition was primarily cohesive. This is also the case for PG58-28 and PG64-22 # 3, where the retained cohesive values are around 80%. Looking at PG64-22 # 1 and PG64-22 # 2, both had very low retained cohesive values (under 30%), which means that the principal mode of failure was adhesive. For the limestone substrate, the principal mode of fail-

Table 6. Results of the ANOVA analysis performed at a 95% confidence level.

Binder	Type of substrate for dry tests		Wet conditioning for glass substrate		Wet conditioning for limestone substrate	
	Significant	p-value	Significant	p-value	Significant	p-value
PG58-28	NO	0.1688	YES	0.0001	YES	0.0337
PG64-22 # 1	Bartlett test negative		YES	0.0370	YES	0.0028
PG64-22 # 2	NO	0.2806	YES	0.0349	NO	0.0537
PG64-22 # 3	NO	0.0982	YES	0.0178	NO	0.1029
PG70-28	NO	0.2379	NO	0.4942	NO	0.0513
PG76-28	NO	0.9151	NO	0.0522	NO	0.9752

ure in wet condition was cohesive (i.e., retained cohesive values higher than 70%), except for PG64-22 # 2. In this case, the retained cohesive value was very low (approximately 35%).

3.5. Measurement of the stress-strain evolution

To explain the increase in pull-off strength observed after water conditioning, the pull-off test apparatus was modified to allow a measurement of the displacement. A plastic head was fabricated to receive two of 12.5 mm LVDTs (Fig. 5). The displacement measurements were done directly on the pull-stub. For each test, the average of the displacement measurements of the two LVDT was considered. Figure 6 presents an example of the relation between displacement and time. There are two distinct displacement evolutions. The rate of increase of the displacement (i.e., the slope of a linear regression) for phase 1 is significantly lower than for phase 2. The transition between phase 1 and phase 2 is characterized by an inflection point. Because the displacement measurements were carried out from a separate unit from the pull-off device, we could not establish the exact moment at which failure occurs. Therefore, the assumption that failure occurs at the transition between phase 1 and phase 2 was made. From the displacement evolution slope of phase 1, the stress-strain relation was calculated.

Figure 7 shows the stress-strain relation for the PG58-28 binder tested with the glass substrate both in dry and wet conditions. Further, the strain amplitude for a stress of 0.8 kPa is indicated. In this case, the strain values for 0.8 kPa in wet condition are lower than those in dry condition. We can assume that PG58-28 is more rigid in wet condition than in dry condition. This observation can be related with the results previously presented; the pull-off strength of the PG58-28 binder with glass substrate is higher in wet condition than in dry condition (Fig. 3) even though stripping occurred (Table 5). Therefore, there is an increase in the binder stiffness due to hot water conditioning.

The displacement measurements were also done on the PG64-22 # 2 binder with glass and limestone substrate in dry and wet conditions. There is also an increase in the binder stiffness for the glass substrate in wet condition as compared to the dry condition, as shown in Fig. 8. The opposite trend is observed for the limestone substrate. There is a decrease in the binder stiffness in wet condition, as compared with the dry condition. This is in agreement with the test results previously presented. For the glass substrate, the pull-off strengths in wet condition are higher than in dry condition. For the limestone substrate, the pull-off strengths in wet condition are lower than in dry condition.

4. Statistical analysis of the test results

To verify the effect of the substrate type as well as the water conditioning on the pull-off strength, a statistical analysis was performed. Using a one-way analysis of variance (ANOVA) with a 95% confidence level, three hypotheses were verified: (1) the type of substrate has an effect on the pull-off strength in dry condition, (2) wet conditioning has an effect on pull-off strength for glass substrate, and (3) wet conditioning has an effect on pull-off strength for limestone substrate. The ANOVA is generally presented as a

statistical test to compare the means of two or more populations. As such, it is possible to determine if there are statistically significant differences between two populations.

Prior to performing the ANOVA analysis, two basic hypotheses must be verified: (1) each population is normally distributed, and (2) the variances of the populations are equal (Bourges and Faur-Brasquet 2003). In this case, because the size of the population is small ($n = 3$ for each binder-substrate combination), it could be assumed that each population was normally distributed. Moreover, small differences from normality of a population do not significantly affect the ANOVA analysis (Ryan 2007). The equality of the variances was verified with the Bartlett test for each of the three hypotheses. One Bartlett test gave a negative result, meaning that this combination cannot be included in the ANOVA analysis (PG64-22 # 1 for dry test). The results of the ANOVA analysis performed are presented in Table 6, where it is indicated whether each effect analyzed is significant; as well, the p-value is shown. For a 95% confidence level, the error margin is 5% (0.05). If the p-value is lower than 0.05, it means that there are significant differences between the population analyzed, and the hypothesis is thus verified.

With respect to the effect of the type of substrate on the pull-off strength, no significant differences between glass substrate and limestone substrate for all tested binders were observed. It is fair to say that in dry condition, the pull-off strength is independent of the substrate type, and thus that the bonding strength is rather affected by the binder type. This observation confirms what has been observed in other research works (Zhang et al. 2017).

Regarding the glass substrate, the ANOVA analysis showed that water conditioning has a significant effect on PG58-28 and on the three PG64-22 binders (all four being neat binders). Both binders modified with polymers (PG70-28 and PG76-28) with glass were not affected by water conditioning, according to the ANOVA analysis. For the binder-limestone substrate combination, the statistical analysis showed that the water conditioning effect is significant for PG58-28 and PG64-22 # 1. In this case, the effect of water conditioning for PG58-28 is expressed as an increase in the pull-off strength and a decrease for PG64-22 # 1.

5. Conclusion

In this research, the pull-off strength of various binders (six) with glass and limestone substrates were measured. All binder-substrate combinations were tested in dry condition and after water conditioning (7 days in a 60° water bath). From the analysis conducted on the test results, the following conclusions were drawn:

- In dry condition, no significant differences were observed between the pull-off strength of glass and limestone substrates. This suggests that in dry condition, the pull-off strength is binder-dependent rather than substrate-dependent. Moreover, all tested binder-substrate combinations in dry condition showed cohesive failure;
- After water conditioning, the pull-off strength values are higher for the glass substrate combination than for the lime-

stone substrate one. A possible explanation for this observation is the difference in terms of moisture absorption. Glass has no absorption, while limestone does;

- Significant differences were observed for the glass substrate between the pull-off strength of the dry condition versus the wet condition for the neat binders (PG58-28 and all three PG64-22). There was an increase in the pull-off strength after water conditioning. On the other hand, the effect of water conditioning was not significant for polymer-modified binders with glass substrate;
- For the limestone substrate, two binders were significantly affected by the water conditioning. In those cases, the effect of water is expressed as an increase in the pull-off strength for one binder (PG58-28) and a decrease for the other one (PG64-22 # 1).

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