RHEOLOGICAL PROPERTIES OF ASPHALT BINDERS WITH CHEMICAL TENSOACTIVE ADDITIVES USED IN WARM MIX ASPHALTS (WMA)

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ABSTRACT

The warm mix asphalts (WMA) have been developed with the objective of minimizing the CO2 emissions in the production and placement process of hot mix asphalts (HMA) by reducing temperatures at which these are mixed and compacted. However this reduction must not affect the manufacture and final performance of asphalt. The WMA additives allow reducing the production temperature, maintaining the mixture workability during the mix process and without compromising the final performance of concrete asphalt. There are different additives, some of them modify the rheological behaviour of bitumen (wax or paraffin) while others in theory maintain unaffected its rheological behavious).

In this work the differences in rheological properties of conventional and polymer modified bitumens, with and without chemical additives, obtained from HMA and WMA were studied by a Dynamic Shear Rheometer (DSR). Additionally tests like moisture damage analysis and Wheel Tracking on air and under water were made in these asphalts. The rheological properties of polymer modified bitumen have been affected by the WMA additives, while in the conventional bitumen the rheological properties have not been significantly affected. The WMA additives improve the workability of asphalt during the production process and improves the performance of WMA tested in the Wheel Tracking under water.

Keywords: Warm Mix Asphalt (WMA), WMA additives, rheology, asphalt performance

1. INTRODUCTION

Due to the importance of environmental question in the manufacture of hot mix asphalts (HMA) new technologies have been developed. The warm mix asphalt (WMA) appears with the objective of minimizing the CO_2 emissions in the production and placement process of HMA by reducing temperatures at which these are mixed and compacted. This allows to minimize the amount of energy required, to reduce the emissions and odors and to improve the welfare of workers. Another important question is maintaining an acceptable pavement performance.

The different WMA technologies look for improve the workability and compactability of asphalts and can be classified in two major types; those that use water and those that use some form of additive incorporated to the bitumen to obtain the temperature reduction [1].

Processes that introduce small amounts of water to hot bitumen, either via a foaming nozzle or a hydrophilic material such as zeolite, or damp aggregate, rely on the fact that when a given volume of water is dispersed in hot bitumen, it results in an expansion of the binder phase and a corresponding reduction in the mix viscosity; then a reduction in temperature is possible [1].

Organic additives; Fischer-Tropsch wax, montan wax, or fatty amides [1]; or chemical tensoactive additives [2] are incorporated to the bitumen. The former ones produce a decrease in bitumen viscosity above the melting point of the wax, at mixing and paving temperatures, meanwhile the other additives reduce the surface tension of bitumen binder without modifying, in theory, the rheological properties.

In this work the rheological properties of conventional and polymer modified bitumens, with and without chemical tensoactive additives, extracted from HMA and WMA were studied. Additionally in this asphalts the rutting and moisture susceptibility performances were evaluated in Wheel Tracking in air and under water respectively.

2. EXPERIMENTAL

2.1 Test program

The rheological properties of bitumen with and without WMA additive were analysed in the Dynamic Shear Rheometer (DSR). The different bitumen binders were extracted from WMA and HMA. The main objectives were to observe the effect in the rheological properties of pure bitumen (without additives) owing to reduce the elaboration temperatures and the effect on the behaviour of bitumen with additives respect to pure one.

The HMA were mixed and compacted at conventional temperatures (T1) according to traditional viscosity values of 0,17 and 0,28 Pa.s respectively.

The WMA were made at temperatures (T2) obtained following the recommendation of Khatri et al. [3], using the Zero Shear Viscosity (ZSV) concept, according to viscosity values of 0,75 and 1,4 Pa.s for mixing and compacting respectively.

HMAs and some WMAs were made with pure bitumen (without additives) while additives were only used to elaborate WMAs.

The bitumen binders for rheological analysis were extracted directly of asphalts after performance tests according to the ASTM D 1856 (2003).

The frequency sweep and Multiple Stress Creep Recovery (MSCR) were the rheological tests procedures selected to study the different bitumen binders meanwhile rutting performances in the Wheel tracking test (WTTair) and moisture damage in submerged wheel tracking test (WTTwater) were evaluated in the different asphalts.

2.2 Materials and asphalts

In this work two commercial bitumen binders in Argentina, Conventional (C) and Polymer Modified (PM), were studied. Table 1 presents their main characteristics including penetration, softening point (R & B), Brookfield viscosity at 60 °C and their Performance Grades (PG) according to ASTM D 6373 standard. Table 1 also shows the mixing and compaction temperatures for T1 and T2 criterion.

Both binders were blended with two different chemical tensoactive additives (A and B) that reduce the bitumen surface tension. The A additive contain surface active agents, it is presented in a liquid form and was added to the bitumen in 0,4 % by weigh of binder. Meanwhile B additive contains resins, polymers and adhesive agent; it is presented in pellets form and was incorporated to the bitumen in 2% by weigh of binder. Both additives were incorporated prior to the elaboration of asphalts. To blend the additives the bitumen was heated for two hours in an oven at a temperature that ensures a viscosity of 0,3 Pa.s (150 and 180 °C for C and PM respectively). In this condition the additives were incorporated and blended by means of a pallet stirrer.

Coarse dense grade asphalt (D-12), see Fig 1, was used to evaluate the different asphalt performances of HMAs and WMAs. Two coarse aggregates (6-20 and 6-12 mm) and one crush sand (0-6 mm) from the province of Buenos Aires in Argentina were used. The Marshall method (ASTM D 1599) was used to design the HMA; the optimal bitumen content

was found in 5 % for both type of binder (C and PM). The WMA were made according to this design in respect to bitumen content.

Bitumen		С	PM			
Modification		-	SBS			
Argentina standard classification		70/100	AM3-C			
Penetration at 25 °C	[dmm]	88	64			
Softening point (R & B)	[°C]	47,6	95,5			
Brookfield Viscosity at 60°C	[Pa.s]	150,8	-			
Torsional Recovery	[%]	-	77,2			
SHRP Performance Grade (PG)		58-22	70-22			
Manufacture temperature. Mixing – Compaction						
T1 (0,17 and 0,28 Pa.s)	[°C]	152 - 140	194 – 184			
T2 (0,75 and 1,40 Pa.s)	[°C]	120 - 109	157 – 149			
ΔΤ	[°C]	32 - 31	37 - 35			

Table 1. Bitumen Properties



Figure 1. Mixture gradation

2.3 Test procedures

2.3.1 Rheological test

A DSR Paar Physica SM-KP with a Rheolab MC-100 was used to evaluate the rheological behaviour of the binders. The equipment has a thermo stabilizer to allow the temperature to be set in a range from 0 to 90 °C through a water recirculation system that surrounds the bitumen sample. The frequency sweep and Multiple Stress Creep Recovery (MSCR) tests were the rheological test selected to characterize the behaviour.

The frequency sweeps were done from 1 to 10 Hz in the temperature range between 10 and 80 °C at 10 °C steps. The Plate-plate configuration, 25 mm diameter and 1 mm gap sample geometry, was used. The frequency sweeps were done inside the linear viscoelastic region of the studied binders. The frequency sweep test results were plotted in a form of Black diagrams [4]; in this way frequency and temperatures were no considering and not was needed any transformation to interpret the data, like master curves. Figure 2 shows typical Black diagrams for conventional and modified bitumens.



Figure 2. Typical Black Diagrams.

The MSCR is based on the repeated creep recovery test [5]. Here the DSR was used to apply a constant stress to bitumen sample for 2 s, and afterwards the load was removed; then the sample was allowed to relax for 18 s. This cycle was applied seven times for 100 Pa stress and then other seven cycles (2 s of load and 18 s of recovery) were applied for 3200 Pa stress. This test methodology is similar to the standard ASTM D 7405, being the only difference the use of total 20 cycles, 1s of load and 9 s of recovery. This test configuration cannot be applied here due to DSR limitations. The test was carried out at a constant temperature of 60 °C; this was chosen here because it is the temperature of Wheel Tracking Test in air and similar to the Wheel Tracking test under water.

During the test the bitumen strain was measured for the fourteen cycles. Then the average of percentage recovery (% cr) for the seven cycles at each stress level and the non-recoverable creep compliance (Jnr) at the end of the test as eq. 1, 2 and 3 indicates were calculated.

$$\% \varepsilon_r (0,1kPa) = \frac{1}{7} \sum_{i=1}^{7} \frac{(\varepsilon_{2s} - \varepsilon_{20s})}{\varepsilon_{2s}} x_i 100$$
(1)

$$\% \varepsilon_r (3,2kPa) = \frac{1}{7} \cdot \sum_{i=8}^{14} \frac{(\varepsilon_{2s} - \varepsilon_{20s})}{\varepsilon_{2s}} \,_i \, x100$$
⁽²⁾

$$Jnr = \frac{Final \, accumulated \, strain}{3,2} \quad \left[\frac{1}{kPa}\right] \tag{3}$$

where % ϵ_{1s} is the average of percentage strain recovery at specified stress, ϵ_{2s} is the strain value at the end of the creep portion after 2 s of load in the i cycle and ϵ_{20s} is the strain value at the end of recovery portion at 20 s in the i cycle. The MSCR is a bitumen test related with rutting behaviour created to complement the performance grade classification of modified bitumen binders in ASTM D 6373. This method was designed to evaluate the elastomeric response of modified bitumen. The percentage recovery (ϵ_{1}) obtained can be used to detect the ability of modified bitumen to maintain elastic response at different stress levels. A decrease of 15 % in ϵ_{1} from 0.1 to 3.2 kPa ($\Delta\epsilon_{1}$) as indicated eq. 4 is consider a limit for determining if the binder has a suitable elastomeric response.

$$\Delta \varepsilon_r = \varepsilon_r \left(3, 2\,kPa \right) - \varepsilon_r \left(0, 1\,kPa \right) \tag{4}$$

The Jnr is a measure of the permanent deformation at the end of the test. When higher is Jnr, weaker is the bitumen resistance to deformation induced by creep-recovery solicitations at high stress levels. High Jnr values are supposed to mean low resistance to permanent deformation. The MSCR is not considered for conventional asphalts; however it was measured here because it represents a good parameter to compare the behaviour of the different conventional binders studied (By reducing temperature and with or without additives). The percentage recovery was not measured in this kind of bitumen.

2.3.2 Asphalt tests

The performance characterization of the asphalt was made in the Wheel Tracking and submerged Wheel Tracking tests (WTTair and WTTwater respectively). Additionally moisture damage analysis was made in asphalts.

The WTTair (B.S. 598 part 110) was used to characterize the asphalt rutting performance under laboratory controlled conditions. The device consists in a solid rubber wheel, 207 mm diameter and 47 mm wide, loaded with 520 ± 5 N. The loaded wheel describes a simple harmonic motion with a total travel distance of 230 mm and a frequency of 21 cycles per minute over an asphalt concrete sample. Test samples were compacted to the design Marshall density. Rutting depth was measured at one minute intervals through a LVDT during 120 minutes. The test temperature was 60 °C. The WTTwater (AASHTO T 324) was used to characterize the asphalt performance under the action of traffic and water. The device consists in a solid steel wheel 203 6 mm diameter and 47 mm wide, loaded with 705 N. The loaded

water. The device consists in a solid steel wheel, 203,6 mm diameter and 47 mm wide, loaded with 705 N. The loaded wheel describes a simple harmonic motion over an asphalt concrete sample with 7 ± 1 % air voids. The wheel travels front and back with a frequency of 25 cycles per minute during six hours. The test temperature was 50 °C. Rut depth was measured at one minute intervals through a LVDT during the test. The rut depth versus wheel passes was plotted. The curve can be divided in three parts. First is produced a rut slope (owing to wheel load) followed by the striping slope if the asphalt has moisture damage. Between slopes appears the striping point which is a measure of mixture moisture susceptibility and is related with the fail in adhesion between aggregate and asphalt.

The moisture damage evaluation was made according to the AASHTO T 283 methodology. Nine asphalt samples, 7 ± 1 % air voids, are divided in three groups. Three specimens are selected as a control and tested without moisture conditioning; three more specimens are selected to be conditioned by saturating with water undergoing a freeze cycle, and subsequently having a warm-water soaking cycle. The final three especimens only suffer the warm-water soaking cycle. The specimens are then tested for indirect tensile strength by loading the specimens at a constant rate and measuring the force required to break the specimen. The tensile strength of the conditioned specimens is compared to the control specimens to determine the tensile strength ratio (TSR); TSR lower than 80 % indicated asphalt that is prone to suffer moisture damage.

3. RESULTS AND DISCUSSION

In this work the rheological properties of conventional and polymer modified bitumen binders, with and without chemical tensoactive additives, extracted from HMA and WMA mixtures were studied. Additionally in these mixtures the Wheel Tracking test in air and under water performances were evaluated. It is important to mention that the WMAs were made following the recommendation of Katri et al. [3]. With this consideration the reduction of temperature results between 30 and 35-40 °C for the C and PM bitumen respectively, see Table 1. The following sections show the finding results.

3.1 Rheological properties of pure bitumen used in HMA and WMA

First the rheological behaviour of pure bitumen from HMA and WMA were analysed. Figure 3 shows the Black diagrams of C and PM bitumen for T1 and T2 manufacture conditions, besides it is shows the Black diagram for the original bitumen as control. In the case of C bitumen a shift to lower values of δ it is observed due to ageing of asphalt, lower for T2 Conditions as expected. In the case of PM asphalt it is observed a minor elastic behaviour in the ageing bitumens, PM(T1) and PM(T2), respect to the original one being similar to conventional asphalt behaviour.



Figure 3. Black Diagrams of bitumens of HMA and WMA without additives; Conventional (left), Polymer Modified (Right).

Figure 4 shows the MSCR test results at 60 °C for C and PM pure bitumen at T1 and T2 temperature conditions. It was observed how the binders of WMA (T2 condition) shown a higher accumulated strain than the binders of HMA (T1 condition) in both types of bitumens being more important in the polymer modified one (PM). This fact can be related with a minor ageing of binder and increased rutting risk in asphalt.

Table 2 shows the calculated parameters obtained in the MSCR tests for the different binders; ε_r (0.1 and 3.2 kPa), Jnr and the $\Delta \varepsilon_r$. It is observed that the Jnr values of PM(T1) are lower, the half, than PM(T2) values; then bitumen PM(T2) is more susceptible to rutting than PM(T1) as was said previously. Considering the ε_r values PM(T1) show a poor elastic response as well as PM(T2); a decrease higher than 15 % was observed in ε_r with the change in stress level, see $\Delta \varepsilon_r$ in table 2. It is important to note that a worse behaviour was observed in PM(T2).

In the cases of C asphalt both Jnr values are similar and high; then mixtures prepared with these particular binders are susceptible to suffer rutting at 60 °C at least. In the case of C this is a conventional bitumen had insignificant and null recover strain for the stress levels of 0,1 and 3,2 kPa respectively.



Figure 4. Pure bitumen MSCR tests.

Table 2. Bitumen (without additives) MSCR tests results.

Bitumen	С	С	PM	РМ
	(T1)	(T2)	(T1)	(T2)
% ε _r (0,1 Pa)	-	-	100	97,7
% ε _r (3,2 Pa)	-	-	22,9	14,8
Jnr [1/kPa]	60,4	68,2	2,6	5,8
$\Delta \varepsilon_{\rm r}$ [%]	-	-	77,1	82,9

Considering the rheological results the reduction of mixing and compaction temperatures does not produce significant changes in the C bitumen behaviour. However the results of PM bitumen without additive reflect that it has increased its rutting risk when used in asphalt. This is due basically to less ageing of the asphalt binder and must be take into account.

3.2 Rheological properties of bitumen binders with and without additives used in WMA

The main objective of this work was to observe if the chemical tensoactive additives used caused some effect in the rheological properties of asphalt binders, then frequency sweep and MSCR tests were done in the asphalts with additives extracted from WMAs. Figure 5 shows the Black diagrams for C and PM bitumens plus additives (C+A(T2), C+B(T2), PM+A(T2) and PM+B(T2)); figure 5 also shows the results obtained for the asphalts without additives, C(T2) and PM(T2), as control.

It can be seen that the incorporation of additives changes the rheological behaviour of bitumens; it is observed an improvement of elastic behaviour of them. In the other hand the effect on the rheological behaviour of bitumens, C or PM, is the same independently of type of additive incorporated; at less for the studied cases.

It is important to note that the incorporation of additive for the C bitumen result in a behaviour similar to a modified bitumen.

In the case of PM bitumen plus additives (PM+A and PM+B), they show a major improvement in elasticity for low G* values (low frequencies or high temperatures), see figure 5-right; then these bitumens may offer a better rutting response.



Figure 5. Black Diagrams of bitumen of WMA (T2); Conventional (left), Polymer Modified (Right).

Figure 6 shows the MSCR tests at 60 °C of C and PM bitumens with additive and also shows the result obtained from the binder without them at T_2 as control. The MSCR tests show that the addition of WMA additives does not significantly change the rheological behavior of C bitumen. However the PM binder with additives (PM + A or PM + B) show drastic changes in their behavior respect to pure one, PM(T_2). In first place it was observed a decrease in the accumulated strains at the end of the test with Jnr values comparable to the bitumen binder of HMA (PM(T_1)), see Table 3. Additionally was observed a significant improvement of the elastic recovery with values of $\Delta \varepsilon_r$ ($\varepsilon_{r3.2 \text{ kPa}} - \varepsilon_{r0.1 \text{ kPa}}$) close to the 15 % of the specification in the case of PM+A bitumen. Despite the PM+B binder not fulfill the specification ($\Delta \varepsilon_r = 32.9$ %) a major improvement was observed in their elastic response respect to PM ($\Delta \varepsilon_r = 82.9$ %). In consequence the incorporation to additives improves the elastomeric response of PM bitumen; this is traduced in a better response to rutting.



Figure 6. MSCR tests results of bitumen binders of WMA.

Bitumen	С	C+A	C+B	PM	PM+A	PM+B
	(T2)	(T2)	(T2)	(T2)	(T2)	(T2)
% ε _r (100 Pa)	-	-	-	97,7	92,3	97,7
% ε _r (3200 Pa)	-	-	-	14,8	74,8	64,8
Jnr [1/kPa]	68,2	55,8	65,3	5,8	1,8	2,3

Table 3. MSCR tests results of bitumen binders of WMA.

3.3 Asphalt performances

The rutting performance and moisture susceptibility of HMAs and WMAs were studied by means of the WTTair and WTTwater; these were selected due to WMAs generally fail in these tests, according to reported by several researches. Fig. 7 and 8 show the permanent deformations measured in the WTTair of the studied asphalts; in table 4 can be observe the main volumetric properties of these asphalts. A worse performance in asphalts with bitumen C was observed when the reduction of temperature was achieved (from T1 to T2), see fig. 7; even with the incorporation of additives (A or B) to the bitumen C the WMA rutting performance was not improved.



Figure 7. Performances of asphalt with C bitumen binders in WTT_{air}.

Mixture		HMA	WMA	WMA	WMA	HMA	WMA	WMA	WMA
(Condition)		(T1)	(T2)	(T2)	(T2)	(T1)	(T2)	(T2)	(T2)
Bitumen		С	С	C+A	C+B	PM	PM	PM+A	PM+B
Bitumen Content	[%]	5,0	5,0	5,0	5,0	5,0	5,0	5,0	5,0
Density	$[g/cm^3]$	2,400	2,399	2,380	2,385	2,398	2,400	2,410	2,403
Air void	[%]	4,4	4,4	5,2	5,0	4,5	4,4	4,0	4,3

Table 4. Volumetric properties of WTTair asphalt samples.

The performances in WTTair of asphalts with PM bitumen binders (PM, PM + A or PM + B) did not show significant changes between them even with the temperature reduction or the absence of additive, see Fig. 8. A possible explanation could be found in the shear stress applied during the test as it was performed here. This shear stress could be not enough to achieve changes in the behaviour of the different asphalts made with PM. D'Angelo et al. [5] said that if the applied shear stress is low, it does not produce enough stress in the modified asphalt to get the polymer chains to slip, and then any differences in the behaviour could not be expressed. If a higher load during more time is applied, e. g. in WTTair standard UNE 12697-22 [16], the differences in the behaviour could be achieved as it was observed in the rheological tests.



Figure 8. Performances of asphalt with PM bitumen binders in WTT_{air}.

Figure 9 and 10 show the permanent deformations measured in the submerged wheel tracking test (WTTwater); it was observed how the temperature reduction and the inclusion of WMA additives to the asphalt produce changes in the performance of mixture. First the temperature reduction produce an important decrease in performance of WMA made with both asphalts, C(T2) and PM(T2), respect to the HMA. The results show that the HMA with C, see figure 9, is asphalt with poor moisture damage resistance and this is significantly diminished when elaborated at reduced temperatures. In the figure can be seen that the addition of chemical tensoactive B to the binder improved the performance of WMA; obtaining a similar performance to the HMA. On the other hand the additive A did not show any improvement of performance to the WMA.



Figure 9. Performances of asphalt with C bitumen binders in WTT_{water}.





Figure 10 shows the performances of asphalt with PM; this accuses a moisture damage (striping slope) when is made at minor temperatures (WMA) that did not present the HMA; the inclusion of additives to the binder (A and B) improves the performance of WMA elaborated with PM bitumen obtaining similar performance as HMA (T1 condition). It is important to mention when plastic deformation occurs (stripping slope appears) this means a failure of adhesion between bitumen and aggregates. The tests results on asphalts with C bitumen (C(T1), C(T2), C+A(T2) and C+B(T2)) shown striping slope. C(T1) and C(T2) asphalts shown materials loss while the other only shown an excessive rutting. In the case of PM(T2) asphalt the failure is an excessive rutting without loss of materials in the surface sample. The WTTwater performance tests show similar results to those observed in rheological tests, most with the MSCR test. This performance test appears as an efficient tool to show the different behaviours due to reduction of mixing and placement temperatures and also shows the positive effect of WMA additives used.

Table 5 shows the results of moisture damage evaluation with the AASTHO T 283 methodology. It is observed how the WMA with pure C bitumen made at lower temperature (T2) shows moisture damage; only one of the additives (B) improves the moisture damage being similar to the HMA result. In the case of PM bitumen all asphalt pass the test and no present moisture damage.

Table 5. M	oisture damage	evaluation	with	AASTHO	Т	283
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	TSR [%]							
	С	C+A	C+B	PM	PM+A	PM+B		
T1	88			89				
T2	54	62	84	82	76	90		

4. CONCLUSIONS

The rheological properties of Conventional (C) and Polymer Modified (PM) bitumens, with and without additives (two different chemical tensoactive additives (A and B)), extracted from HMA and WMA were studied. Additionally different tests on asphalt were evaluated. The main conclusions are indicated as follows.

The rheological properties of pure conventional bitumen (C) from WMA did not show significant changes respect to bitumen from HMA. However the addition of the WMA additives at bitumen improves its elastic behaviour measured in the frequency sweep test.

The rheological properties of the PM bitumen without additive accuse different behaviour when decreasing the manufacture temperatures; the results represent the behaviour of bitumen that increment the rutting risks if it is used in asphalts. The differences became clearer when was observed in the Multiple Stress Creep Recovery (MSCR) test. This is due basically to a less ageing of the binder and must be take into account.

The additives for WMA modified the rheological behaviour of the PM bitumen; the PM with additives (A or B) shows improvement respect to the pure bitumen from the WMA. These bitumens present a more elastic behaviour to low frequencies in the frequency sweep test. Besides they show a significant reduction of the accumulated strain in the MSCR test, being similar to PM asphalt extracted from HMA. The elastic response of modified asphalt with tensoactive additives has been improved.

From the performance tests it was observed a worse rutting behaviour in the Wheel Tracking in air for the C asphalt with temperature reduction; even the WMA performance was not improved when the additives were incorporated to the bitumen.

In contrast to rheological measures, the rutting performance tests in asphalts with PM bitumen did not show significant changes among the studied cases, either by temperature reduction or with additives incorporation. A possible explanation could be found in the load applied during the test. This load could not produce enough shear stress in the modified binder to get the polymer chains to slip, being less possible the occurrence of differences on behaviour. Respect to moisture susceptibility of mixtures studied in the submerged Wheel Tracking test, it was observed an important performance decrease in the mixtures made with both asphalts with the reduction of mixing and compaction temperature. The inclusion of additives to the binder improves the performance of WMA in the Wheel Tracking under water elaborated with PM bitumen, being similar to the HMA performance, while in the C asphalt only one of the additives improves the performance.

Respect to moisture damage evaluation with the AASTHO T 283 it was observed that the incorporation of additives improves the moisture damage in the WMA with C bitumen; while the PM asphalts pass the test in all studied cases.

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