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PERFORMANCE OF BITUMEN 70/100 OBTAINED FROM DIFFERENT SUPPLIERS

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Performance of bitumen 70/100 obtained from different suppliers

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ABSTRACT. Bitumen in Europe is produced according to the European bitumen specifications (EN 12591). Even if these specifications are met, the performance of a bitumen can substantially vary depending on the supplier. This means that the current specifications for penetration grade bitumen have little correlation with the functional properties of asphalt concrete.

In this study 5 different deliveries of bitumen 70/100 at 4 different asphalt mixing plants were anonymously collected and its functional properties were tested.

The performed laboratory tests show that especially the fatigue life of the tested bitumen samples vary significantly. Correlations with functional asphalt characteristics indicate that this has influence on the performance of asphalt concrete in the field.

KEYWORDS: bitumen tests, fatigue, cracking, specifications, asphalt performance.

1. Introduction

As from 2008, functional asphalt concrete tests are specified in The Netherlands. From obtained test results, there is some concern about the variation in quality of the used bitumen. To verify the quality and the influence of penetration grade bitumen from different suppliers a pilot study was performed with regular used bitumen 70/100 for pavements by the Dutch Asphalt Pavement Association (VBW-Asfalt). For the purpose of this, samples of bitumen were collected at different asphalt mixing plants during unloading of bitumen from a bitumen truck according to an equal sampling protocol (EN 58). The obtained samples of bitumen were labelled A to E and tested anonymously in a laboratory for their (functional) properties.

In this paper the results are presented of the performed study in relation with functional asphalt behaviour. Because of the limited time and available budget, this (limited) study concerned in particular the equality within the bitumen 70/100 grade and in the course of time (several batches from 1 supplier).

2. Experimental plan

In total 5 bitumen samples of penetration grade 70/100 from 4 different bitumen suppliers were tested. The performed tests on the bitumen are given below. The selection of these tests has been based on recent scientific knowledge (Santagata et al., 2009; De Jonghe et al., 2004). The tests were performed under strictly controlled conditions. All supplied samples were reheated for maximum 1 time and the tests were conducted with the same equipment and technician to obtain the highest possible accuracy and repeatability.

Determination conventional properties.

– Penetration (EN 1426)

– Ring & Ball Softening Point (EN 1427).

Stiffness.

– Determination of the master curve by means of frequency sweeps (0.01 to 50 Hz) with a dynamic shear rheometer (DSR) according to EN 14770 at temperatures between -10 and 60 °C after RTFOT ageing (EN 12607-1).

Fatigue.

– Determination by means of a DSR on the basis of the RILEM test protocol in triplicate (RILEM TC 182, 2008) at 10 °C, 10 Hz and a (shear) strain level of 1%, after RTFOT aging. For this strain level was chosen because of the test duration (each sample) of approximately 24 hours, giving the best possible scientific understanding.

Bitumen composition.

– Analysis of the determination of the 4 main fractions of the bitumen (saturates, aromatics, resins and asphaltenes) (Read et al., 2003). In this respect the fraction of asphaltenes is determined according to ASTM D3279 with n-heptane and the other fractions by thin layer chromatography (Iatroscan) with the use of n-pentane and toluene / chloroform.

Rutting.

– Determination of zero shear viscosity (ZSV) at 40 °C in duplicate according to draft standard prEN 15325, carried out at the following conditions: 2 mm gap; shear stress (τ) 30 Pa and a maximum loading time of 4 hours.

Ravelling resistance / adhesion.

– Determination of the acid number, by means of a potentiometric titration method based on ASTM D 664.

– Analysis of the cohesive properties of the bitumen film under high frequency "shock loading" conditions versus temperature with the modified Vialit plate shock test using Moraine gravel after 30 days aging at 70 °C (based on EN 12272-3 and (Goos, 2001)). Per bitumen 6 test plates were made, which were tested in duplicate at 3 temperatures. Stone loss was determined after 1 steel ball dropping.

Asphalt characteristics.

- Performing of 4-point bending tests with 2 selected bitumen's in a specific Dutch asphalt mixture (AC 11 surf 70/100) according to European standard EN 12967-24. Test conditions: Stiffness at 8 Hz and 20 °C and fatigue at 30 Hz and 20 °C. Beam dimensions: 450 x 50 x 50 mm.

3. Test results

3.1 Conventional properties

The main properties according to EN 12591 are displayed in table 1. As expected all the bitumen samples complied with the European standard.

Table 1. Properties according to EN 12591.

Bitumen sample		A	B	C	D	E	Specification
Penetration	[0.1 mm]	91	81	77	84	77	70 - 100
R&B	[°C]	44.4	46.2	46.0	45.4	46.2	43 - 51
PI	[-]	-1.3	-1.0	-1.2	-1.2	-1.2	-1.5 - +0.7

3.2 Stiffness

The master curves of the bitumen samples have been determined after RTFOT ageing by performing frequency sweep tests at temperatures between -10 and 60 °C. The obtained curves using the sigmoidal-model (Medani et al., 2004) are presented in figure 1. From the figure it can be seen that the curves overlap each other most of the time. Bitumen sample A has the lowest stiffness (as expected).
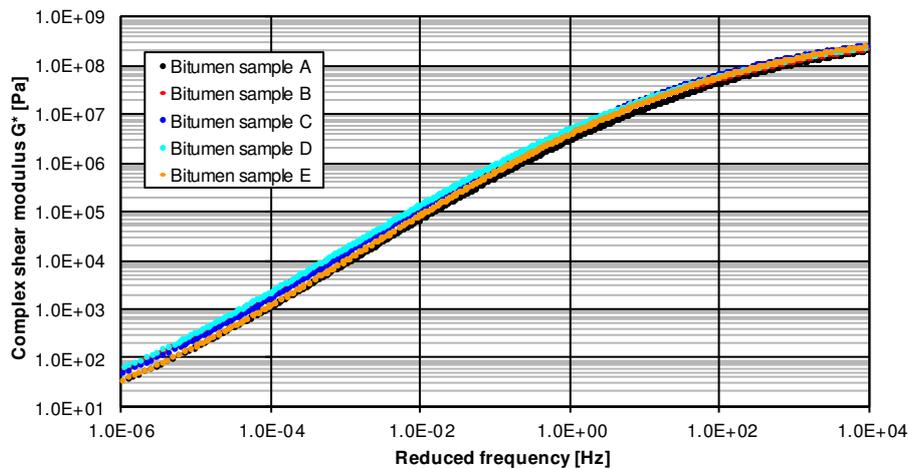

Figure 1. Master curves bitumen samples at a reference temperature of 20 °C.

More in detail, in table 2 the complex shear modulus G* has been determined at a frequency of 1.3 Hz and a temperature of 0, 20 en 40 °C and compared with the penetration. The DSR test frequency of 1.3 Hz ($\approx$ 8 rad/s) corresponds to the asphalt loading frequency of 8 Hz (Bari et al, 2006). This frequency is used for the stiffness determination of asphalt in the Dutch CE-marking (4-point bending configuration).

Table 2. Complex shear modulus G* at 1.3 Hz and 3 different temperatures.

Bitumen sample	0 °C [MPa]	20 °C [MPa]	40 °C [MPa]	Penetration [0.1 mm]
A	81	2.9	0.064	91
B	104	4.5	0.092	81
C	132	4.6	0.076	77
D	115	5.0	0.086	84
E	110	3.9	0.065	77

The results in the table show also for bitumen sample A the lowest stiffness. This is in accordance with the higher penetration value. It looks as if the penetration predicts the difference in stiffness. However, this is not consistent for all samples.

3.3 Fatigue

The fatigue characterization of the bitumen samples has been determined on the basis of the RILEM test protocol in triplicate at 10 °C, 10 Hz and a (shear) strain level of 1%, after RTFOT aging. The average curve (n=3) of the fatigue data is displayed in figure 2 for each bitumen sample.
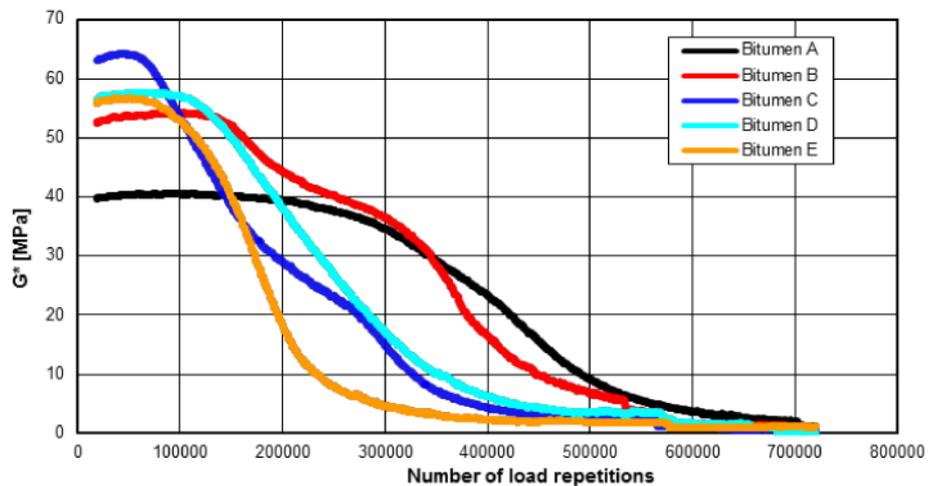

Figure 2. Average (n=3) fatigue curves at 10 °C and a frequency of 10 Hz.

According to RILEM, the fatigue life can be defined in different ways. One definition is the number of load repetitions until halving of the stiffness. Another method is based on dissipated energy ratio (DER). The fatigue life with the latter method can be obtained from the curve in which the number of load repetitions (N) times G* is plotted against N. The fatigue life is at the maximum of N x G* (figure 3).
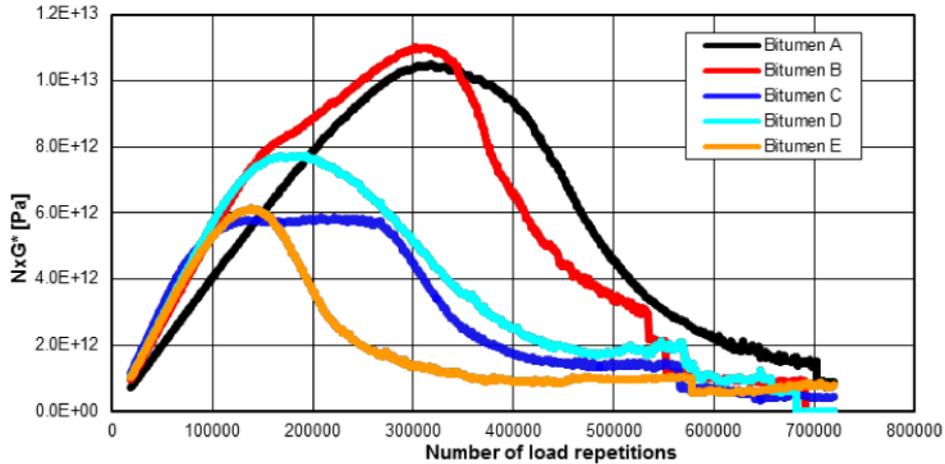
Figure 3. Average (n=3) dissipated energy ratio of bitumen samples.

The results of both methods of defining fatigue life are displayed in table 3. In this table $N_{50\%}$ is the number of load repetitions until halving of G^* and N_{DERmax} the number of load repetitions until the maximum of $N \times G^*$. Due to the strange curve of bitumen sample C, here the centre of the flat part has been used.

Table 3. Fatigue parameters (average lifetimes) tested bitumen samples.

Bitumen sample	$N_{50\%}$	N_{DERmax}
A	427000	319000
B	364000	301000
C	191000	195000
D	248000	184000
E	179000	139000
Average	281800	227600
Coefficient of variation	38.8%	34.4%

From the results it can be seen that bitumen samples A and B clearly show better fatigue properties than the other bitumen samples. From the obtained results, it seems as if there is some correlation with the penetration of the bitumen samples as shown in figure 4. However, adding additional test data of additional suppliers of bitumen 70/100 from work commissioned by an asphalt mixture production plant leads to almost no relationship ($R^2=0.36$).
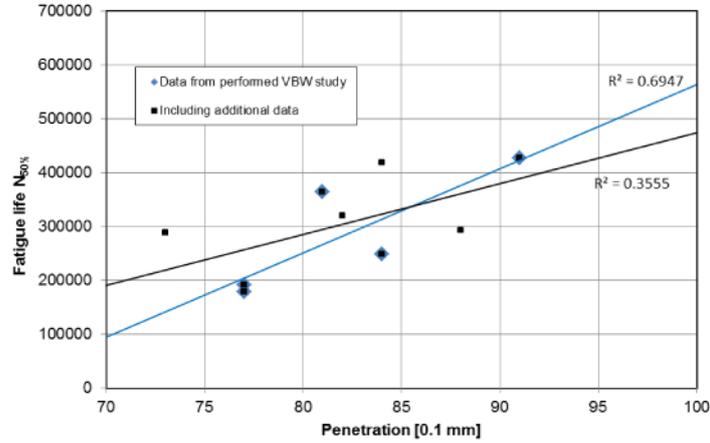
Figure 4. Fatigue life bitumen against penetration bitumen.

3.4 Bitumen composition

The chemical composition has been characterised according to the "SARA" method, in which bitumen is divided in Saturates, Aromatics, Resins and Asphaltenes (Read et al., 2003). The method is not completely standardised. This means that results are not easy to compare with results from other laboratories, due to differences in solvents and procedures.

The method is suitable as a comparison between samples, which are examined in the same laboratory and it gives an insight into the microstructure of the different bitumen samples. The results of the tested samples are shown in Table 4.

Table 4. SARA-analyses bitumen samples

Bitumen sample		A	B	C	D	E
Saturates	[%]	5.6	7.8	5.3	5.5	6.2
Aromatics	[%]	65.7	63.4	69.2	67.9	69.1
Resins	[%]	18.8	17.6	17.0	18.2	15.5
Asphaltenes	[%]	9.9	11.2	8.5	8.4	9.2

In the literature it has been demonstrated (Santagata et al., 2009), that the fatigue- and healing characteristics of a bitumen could be correlated to the microstructure of a bitumen. These characteristic parameters are calculated from the ratios of the SARA fractions. The ratios between the various fractions are listed in Table 5. CII is the colloidal stability index which is defined as:

[1]
$$CII = \frac{Asphaltenes + Saturates}{Resins + Aromatics}$$

Table 5. Microstructure parameters calculated from the fractions.

Bitumen sample	As/R*	S/Ar*	CII
A	0.527	0.085	0.183
B	0.636	0.123	0.235
C	0.500	0.077	0.160
D	0.462	0.081	0.161
E	0.594	0.090	0.182
Average	0.544	0.091	0.184
Coefficient of variation	13.0%	20.2%	16.5%

*) As/R = Asphaltenes / Resin ratio; S/Ar = Saturates / Aromatics ratio.

The numbers show that the parameters do not vary significantly with respect to each other. Only bitumen sample B shows a relatively high S/Ar and CII ratio.

3.5 Rutting

Several studies have shown that there is a correlation between the zero shear viscosity (ZSV) and the rutting sensitivity of an asphalt mixture (Van Rooijen, 2004). The results of the tested bitumen samples are given in Table 6. For comparison, the conventional properties from Table 1 are also given.

Table 6. Zero shear viscosity bitumen samples at 40 °C.

Bitumen sample		A	B	C	D	E	Avg.	COV
ZSV	[Pa.s]	4555	5080	5460	4545	5435	5015	9.0%
Penetration	[0.1 mm]	91	81	77	84	77	82	7.1%
R&B	[°C]	44.4	46.2	46.0	45.4	46.2	45.6	1.7%

Remarkable is the difference between the ZSV value of bitumen samples C and D, while these bitumen samples came from the same producer. Furthermore, there is not much difference between the tested bitumen samples. The rutting characteristics in hot mix asphalt for these binders are expected to be similar.

3.6 Ravelling resistance / adhesion

The durability of an asphalt mix is mainly determined by the adhesion of the bitumen to the mineral aggregate. A bitumen with a poor affinity with the aggregate will therefore, for example cause stripping or early ravelling. The affinity between bitumen and aggregate is partly determined by the (chemical) attraction at the interface. For example an acidic bitumen (high acid number) will not give an optimal bonding with chippings of acidic nature, such as for example granite. What the optimal acid value should be, depends on the application. For an alkaline type of

mineral aggregate a high acid value of the bitumen is favourable. The acid values of the tested bitumen samples are given in table 7.

The durability of the aggregate – binder bonding was further tested with the modified Vialit plate shock test using Moraine gravel (Goos, 2001). This test measures the cohesive and adhesive properties of a bitumen film under high frequency "shock loading" conditions versus temperature. The results obtained in this test are in particular of importance for the application of the bitumen in surface dressings and in porous asphalt. Moraine gravel has been chosen, because this type of mineral aggregate is generally used and has a (light) acidic character. The results are also given in table 7.

Table 7. Acid value and Vialit test results for the tested bitumen samples.

Bitumen sample	Acid value [mg KOH/gram]	50% stone loss [°C]
A	0.30	16.5
B	0.35	20.0
C	0.22	19.5
D	0.33	20.0
E	0.35	18.8
Average	0.31	19.0
Coefficient of variation	17.5%	7.7%

The results show that the acid number for all the 5 bitumen samples is roughly similar, and is quite low. So, the adhesion behavior of the investigated bitumen samples is expected to be similar. Remarkable is the difference between the acid values of bitumen samples C and D, while these bitumen samples came from the same producer. This finding is however not confirmed by the Vialit plate shock test. The Vialit results show the lowest temperature for bitumen sample A. However, the obtained temperatures are quite high in comparison with the values reported in the description of the test (Goos, 2001).

4. Binder properties and correlation with functional asphalt characteristics

The conventional properties according to EN 12591 of the tested bitumen samples appear to show little variation (between different suppliers and deliveries). Based on these properties, it would be (a priori) expected that these bitumen samples will behave similar in an asphalt mixture. However, the functional properties of the tested bitumen samples show some important differences.

The bitumen stiffness has an influence on the stiffness of the asphalt concrete (Bari, 2006). A stiffer bitumen will therefore also give a stiffer asphalt mixture. Indicative stiffness calculations with the calibrated Witczak model (Bari, 2006) for a

Dutch standard AC 22 base mixture at a frequency of 8 Hz and input variables the complex shear modulus G* and the phase angle δ at 1.3 Hz and 20 °C show the influence on the stiffness (see table 8).

Table 8. Calculated asphalt stiffness E* at 8 Hz and 20 °C (indicative).

Bitumen sample	Complex modulus G* [MPa]	Phase angle δ [°]	Stiffness modulus E* [MPa]
A	2.9	60.4	8125
B	4.5	56.3	10000
C	4.6	59.6	9750
D	5.0	55.8	10500
E	3.9	61.1	9000
Average	4.2	58.6	9475
Coefficient of variation	19.5%	4.1%	9.8%

Research commissioned by the Transportation Research Board (TRB) in the USA (NCHRP report 459b) shows a good correlation between the fatigue life of a bitumen and an asphalt mixture. This means that a bitumen with a higher fatigue life in the (same) asphalt mixture will also perform better. The exact correlation depends on the type of aggregate, the grading and the test conditions. In the TRB study this relationship was found to be:

[2] $$N_{50\%, \text{asphalt}} = 0.20111 \cdot N_{50\%,\text{bitumen}} + 10620$$

Using this relationship the fatigue parameters (number of load repetitions) were calculated for asphalt concrete. The results are shown in table 9. From these results it can be seen that bitumen sample A and B give the best results. Asphalt fatigue tests with these bitumen samples will show a better fatigue curve (the curve moves to the right), resulting in a better fatigue strain level (ε_6) in 4-point bending tests according to European standard EN 12967-24.

Table 9. Calculated fatigue parameters asphalt at 10 Hz and 10 °C (indicative).

Bitumen sample	$N_{50\%, \text{bitumen}}$	$N_{50\%, \text{asphalt}}$
A	427000	96494
B	364000	83824
C	191000	49032
D	248000	60496
E	179000	46619
Average	281800	67293
Coefficient of variation	38.8%	32.7%

Additionally performed 4-points bending tests on a specific mixture give an indication on the difference in fatigue strain level (ε_6). The 4-point bending tests were carried out with bitumen obtained from the supplier of sample A and the supplier of sample C and D. The mixture was an AC 11 surf 70/100 without recycled material. The test conditions were 30 Hz en 20 °C according to the Dutch standard (CROW, 2010). The results are summarized in figure 5.
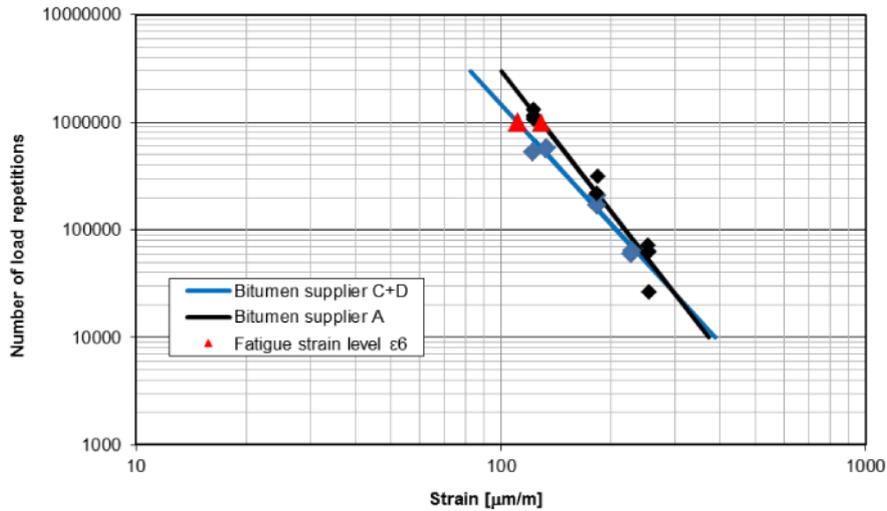

Figure 5. Results fatigue tests asphalt for different bitumen suppliers.

Despite the limited data set (8 instead of 18 specimens per curve), the graphs clearly show the difference between 2 different suppliers of bitumen. The parameter ε_6 was found to be 111 μm/m (bitumen supplier C and D), respectively 129 μm/m (bitumen supplier A). The measured average stiffnesses (8 Hz, 20 °C) were respectively 4115 MPa for bitumen supplier C and D and 3776 MPa for bitumen supplier A. Given the tight ranges for ε_6 in the Dutch specifications an asphalt mixture could because of this easily swift to another classification or will not pass the specifications.

As demonstrated in literature (Santagata et al., 2009), the fatigue- and healing characteristics of a bitumen can also be correlated to its microstructure (composition). According to this study, a high Saturates / Aromatics ratio (S/Ar) should be favourable for the healing characteristics of a bitumen expressed in the so-called Relative Healing Index (RHI). Using the relation between the S/Ar ratio and the RHI as given in equation 3, the values of RHI were calculated for the tested bitumen samples as shown in table 10.

[3] $$S/Ar = 0.126 \cdot RHI + 0.070$$

Table 10. Calculated values for RHI

Bitumen sample	A	B	C	D	E
RHI	0.121	0.421	0.052	0.087	0.157

The table shows that the capability for healing of bitumen sample B seems to be the highest and for bitumen sample C seems to be the lowest.

Furthermore, the chemical composition is also reflected in the colloidal stability index (CII), which has a correlation with the fatigue life of a bitumen (Santagata et al., 2009). A high value should give a better (bitumen) fatigue life than a lower value. The performed DSR fatigue tests show that this correlation is not always valid. For bitumen sample B (relative high CII) the theory is true, but for bitumen sample A (low CII) it is not confirmed (see tables 5 and 9).

5. Conclusions and recommendations

From the results of this (limited) study, it can be concluded that different deliveries of bitumen, which are apparently equal (according to the traditional specifications currently part of EN 12591), can vary considerably in performance when applied in asphalt mixtures.

Functional tests suggest that especially the fatigue life of a bitumen can vary significantly. Correlations with functional asphalt properties show that this has influence on the performance of an asphalt pavement. CE-marking tests of asphalt seems to confirm this finding. To validate the (theoretical) results of the functional asphalt properties, more 4-point bending fatigue tests on (different) asphalt mixtures have to be carried out.

The traditional specifications according to EN 12591 lack an (easy to apply) specification for the fatigue life of bituminous binders. DSR fatigue testing e.g. according to the RILEM test proposal should be included. To validate the findings testing of bitumen from more exotic sources is also advisable.

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