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# Performance analysis of Lignin as an Antioxidant in Bituminous Mixture

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# ABSTRACT:

The present work involves the determination of various rheological properties of bitumen used of grade 60/70 grade. The samples were prepared with two types of lignin, lignin 1 (organic lignin) and lignin 2 (Lignosulfonates). These are blend with different proportions and different laboratory test to enhance the required engineering properties and to arrest its oxidative aging resistance. The short-term aging is conducted with the rolling thin oven test (RTFOT) and its rheological test is done by rotational viscometer. Further the FTIR test is conducted to determine its oxidation property. The optimum binder content is determined for the DBM mix. Using the OBC the indirect tensile strength and the tensile strength ration is determined. The fatigue life cycles is also evaluated.

Keywords: Lignin, Bitumen, Tensile Strength and Rheological Test

# Introduction:

Bitumen is extensively used for the construction of highway and airport pavements, were approximately 85% of the global consumption of bitumen. Bitumen is extremely sticky, completely dissolvable in carbon-disulphide and it is combined polycyclic hydrocarbons. Rough bitumen is a sticky in nature and substantially bitumen must be warmed or drained before it will pour out. It is like chilly molasses at room temperature and its boiling point rises up to 525°C.

Wood is utmost largely available resources in the bio-based industry and it is the most compound resources, made out of polymers of lignin and carbohydrates that are physically and synthetically bound together. Wood species are divided into two groups: hardwood and softwood. Softwoods are gymnosperm trees and hardwoods are angiosperm trees.

Lignin is one of the important chemical constituents of lignocelluloses materials in wood and it is one of the most abundant biopolymers in nature. Lignin, the Latin term lignum significance wood, is an essential part of the secondary cell walls of plants. Lignin clears the cavity in the cell walls amongst cellulose, hemicelluloses and pectin constituents developing mechanical strength to the cell walls. Lignin is the second richest natural substance in the world next to cellulose. It is calculated that  $5 \times 10^6$  metric tons of lignin are manufactured yearly from pulp and paper industries globally. Lignin is a hydrocarbon and contains mainly of carbon, hydrogen, and oxygen. The chemical structure of lignin is extremely aromatic in nature with methoxyl and hydroxyl groups. Lignin can also contain aromatic hydrogen atoms, carbonyl groups, and aliphatic double bonds.

The antioxidant effects of lignin are derived from the scavenging action of their phenolic structures on oxygen containing free radicals. Structures are benzene rings with attached hydroxyl groups. Benzene rings are six carbon structures with each carbon sharing a single and double covalent bond to another carbon. In a phenolic group, there can be one or more hydroxyl groups attached to the benzene ring. The ability of phenolic compounds to be antioxidants is the functional group's ability to neutralize free radicals. Free radicals are known to actively break down substances by breaking apart their chemical structures. A phenol can neutralize a free radical by donating either a proton or an electron. Because of its structure, a phenol is able to do both while remaining relatively stable. Lignin contains a large amount of phenolic groups, making it an effective antioxidant.

# Application of Lignin

Lignin as binder Lignosulfonates is a very influential and sparing cement, going about as a coupling operator or compacted materials. Lignosulfonates applied on unpaved streets reduce environmental impact from flying clean particles and balance out street surface. The coupling capacity makes it a valued part of: Coal briquettes, Biodegradable Plastic, Plywood and molecule board, Earthenware production, Creature bolster pellets, Carbon dark, Fiberglass protection, Manures and herbicides tile glue, clean suppressants, and Soil stabilizers.

Lignin as dispersant Lignosulfonates helps in bunching and settling of undissolved elements after suspensions. By joining the molecule surface, it shields the molecule from being pulled in to different particles and reduces the measure of water expected to utilize the item successfully. The scattering property makes lignosulfonate used in: Leather tanning, Cement mixers, Concrete admixtures, Clay and ceramics, Dyes and pigments, Pesticides and insecticides.

Lignin as an emulsifier Lignosulfonate balances out emulsions of non-soluble fluids, for example, water and oil, making them deeply impervious to breaking. Lignosulfonates are grinding away as emulsifiers in: Pesticides, Asphalt emulsions, Wax emulsions, Pigments and dyes.

# Need for Study

As of due to the varying temperature and atmospheric reaction there may be cause of deterioration in asphalt pavements. These deterioration causes due to the effect of the long-term and short-term aging. These aging effects the asphalt to convert hard and brittle, further leads to pavement failure and deterioration due to increase in density of traffic, moving loads and an insufficient maintenance, to achieve desired engineering properties of bitumen. It is modified with a polymer lignin which is good binding material and acts as an antioxidant to the asphalt by enhancing the desired engineering properties of the modified asphalt and its performances are studied.

#### **Objectives of the Study**

The objectives of the study are as follows:

- To determine the basic properties of the aggregates, bitumen used in the present study.
- To study the rheological properties and performance of lignin 1 and lignin 2 modified both unaged and aged asphalt binder using different laboratory tests.
- To evaluate the aging index of the modified binder by using the antioxidant lignin 1 and lignin 2.
- To determine the Optimum Binder Content for the DBM mix.
- To determine the tensile strength characteristics and to evaluate the fatigue life cycle by applied repetitive load.
- To study the oxidation characteristics using Fourier Transform Infrared (FTIR) spectroscopy investigations.

#### Scope of the Study

The present work involves the determination of various rheological properties of bitumen used of grade 60/70 grade. The samples were prepared with two types of lignin, lignin 1 (organic lignin) and lignin 2 (Lignosulfonates). These are blend with different proportions and different laboratory test to enhance the required engineering properties and to arrest its oxidative aging resistance. The short-term aging is conducted with the rolling thin oven test (RTFOT) and its rheological test is done by rotational viscometer. Further the FTIR test is conducted to determine its oxidation property. The optimum binder content is determined for the DBM mix. Using the OBC the indirect tensile strength and the tensile strength ration is determined. The fatigue life cycles is also evaluated.

#### **Material Used:**

# Aggregates

The aggregates is most important in the construction of the pavements. The aggregates should be strong to withstand the wheel load applied on them, and should resist the wear load and weathering action. The aggregates forms the 90% of the weight to the mix volume.

MORTH V Revision	% passing	by weight	Cumulative retain %	Individual retain %	% of coarse aggregate, fine	Individual weight of 1200gm
Sieve mm	Grading	Blend			aggregate, filler	
37.5	100	100	0	0		0
26.5	90-100	95	5	50		60
19	71-95	83	17	12		144
13.2	56-80	68	32	15		180
9.5	-	-	-	-	54%	-
4.75	38-54	46	54	22		264
2.36	28-42	35	35	11		132
1.18	-	-	-	-		-

Table 1 Aggregate gradation of Dense Bituminous Macadam as per MORTH 2013

0.6	-	-	-	-		-
0.3	7-21	14	86	21	41%	252
0.15	-	-	-	-		-
0.075	2-8	2-8	95	9		108
Filler					5%	60
Total weight of aggregates for sample				100	1200gms	

# Aggregate gradation

Aggregate gradation which describes the properties of dense bituminous mix of the pavement material. It helps in the improve of the durability, stability, fatigue resistance, workability and permeability. For the present work for DBM mix aggregate gradation of grade II is selected according to MORTH (5th Revision 2013). The nominal size of the aggregate is 26.5mm is tabulated in table 1

#### **Coarse aggregates**

The aggregates which retain on 4.75mm sieve are known as coarse aggregates. In these study nominal size of aggregate is 26.5mm. To calculate the basic properties of aggregates tests were conducted and checked with IS recommendations. Aggregates must be clean, durable, hard and uniform quality throughout the mix. The tests are conducted on the aggregate and are tabulated below in table 2.

# Table 2 Physical Properties of Coarse Aggregate

Tests	Test methods	Results	MORTH specifications (5th
			Revision 2013).
Los angles abrasion value, %	IS: 2386 (III)	14.8%	35% maximum
Water absorption, %	IS: 2386 (IV)	0.44%	2% maximum
Impact value, %	IS: 2386 (IV)	19.59%	27% maximum
Specific gravity	IS: 2386 (III)	2.76	2.5-3
Flakiness index and	IS: 2386 (I)	30.15%	35 % maximum
Elongation index, %			



# Figure 1 Coarse aggregate

### Fine aggregate

The aggregates which passing 4.75mm sieve and retained on 0.075mm are known as fine aggregates. Fine aggregates fills the voids in coarse aggregates and helps in stiffening of binder. The stone crusher chips is used of varying sizes. The specific gravity of the fine aggregate is 2.61.



Figure 2 Fine aggregate

The materials sieved through 0.075 mm and retained in pan is the filler material. The main importance of filler is to fill the minute voids and increase in stability and density in the mix of asphalt. In present work stone dust is used as filler materials as shown in figure. The specific gravity of stone dust 2.5. The properties of the stone dust is shown in table 3

### Table 3 Properties of Filler Material as Per MORTH Specification

IS sieve (mm)	Percentage finer				
	As per MORTH	STONE DUST			
0.6	100	00			
0.3	95-100	00			
0.075	85-100	100			
Specific Gravity		2.5			



Figure 3. Stone dust

# 3.2.2 Bitumen

Bitumen is widely used in pavement construction and it is the raw material used in filling the voids and binding the aggregates together. Bitumen selection is done on the basis the site situation, temperature, type of roads and type of traffic and soil constitutions. The bitumen is collected at the Ninthilkallu plant, Bellare, D.K. The grade of the bitumen is 60/70 based on the penetration value. In this study the basic test were conducted on the bitumen like the penetration, ductility, softening point and specific gravity. The properties of the bitumen is tabulated below in table 4.

### Table 4 Physical properties of bitumen

Test	Test methods	Result
Penetration at 25°c, 1/10mm	IS 1203-1978	66
Softening point ( R&B), <sup>0</sup> c	IS 1205-1978	52
Ductility at 25 <sup>°</sup> c, cm	IS 1208-1978	63.4
Specific gravity at 25%	IS 1202-1978	1.02
Flash point, <sup>0</sup> c	IS 1448 ( P: 69)	330
Fire point, <sup>0</sup> c	IS 1448 (P: 69)	350
Brookfield viscometer at 135°c , Cp	ASTM D 4402	152

# 3.2.3 Lignin

Lignin is a Latin word termed lignum meaning wood. Lignin is obtained from the hard wood and soft wood. Lignin is 5x10<sup>6</sup> tons waste produced worldwide from the pulp and paper industries. The two types of lignin used in the present study are collected from Paari Chem Resources, Mumbai. The lignin used in the present work is two types lignin 1 (organic lignin) and lignin 2 (Lignosulfonates). The properties of these two types of lignin are tabulated below in table 5.

# Table 5 Properties of Lignin 1 AND Lignin 2

Properties	Lignin 1 ( Organic )	Lignin 2 (Lignosulfonates )
Colour	Light brown	Light yellow
Hydrogen ion value, Ph	4.50	4.92
Specific gravity	1.12	1.11
Breaking point of dried up samples at	0.68	0.71
stretching, MPa		



# Methodology:

The binder used for these study is VG30 (60/70) grade. The binder physical properties were conducted before blending process. The lignin, used as binding material for asphalt modifying with proportion of 2%, 4% and 6% by weight of an asphalt binder. The blending of lignin powder and asphalt was done with regular mixing at around 110°c to 140°c with continuous stirring so that the during addition of lignin to asphalt there forms a bubbles floating at the begging of mixing stage on the surface should see that bubbles disappears with continuous stirring. The blending process has to be continued for several minutes until the blend with lignin and asphalt mixed properly and no bubbles has to float on the surface even after reheating.

# **Physical properties**

The virgin asphalt binder and lignin 1 and lignin 2 modified asphalt binders has to undergo several physical properties like penetration test, softening point, Viscosity test, Marshall Stability test, Indirect tensile Strength Characteristics, Tensile strength ratio, Fatigue test and FTIR Analysis-Fourier Transform Infrared Spectroscopy.

# Results

# General

In this chapter analysis of test results are given in the table form or in the graphical representation form. The two types of lignin is used as the binder for bitumen (lignin 1 and lignin 2). The physical properties of bitumen like penetration, softening test and rotation viscometer is evaluated and the Marshall properties for the control mix to obtain the OBC, the Indirect tensile test and the tensile strength ratio is evaluated later on fatigue life is determined based on the tensile strength ratio value. FTIR analysis is analysed.

#### **Physical Properties of Bitumen**

In the physical properties of bitumen the penetration value, softening test and the rotational viscometer is evaluated.

#### Penetration value and softening test

### Table 6: Results Obtained for the Penetration Test for Aged and Unaged Modified Bitumen

	Penetration values					
Percentage lignin %	Virgin bitumen (unaged)	Lignin 1 modified bitumen (unaged)	Lignin2 modified bitumen (unaged)	Virgin Bitumen (aged)	Lignin 1 modified bitumen (aged)	Lignin 2 modified bitumen (aged)
0	66			50		
2		58	60		54	52
4		52	54		42	44
6		40	46		38	38



Figure 5: Penetration Test For Aged And Unaged Modified Bitumen

Table 7: Result Obtained For The Softening Point Test For Aged And Unaged Modified Bitumen

	Softening Point values					
Percentage	Virgin	Lignin 1	Lignin2	Virgin	Lignin 1	Lignin 2
lignin %	bitumen	modified	modified	Bitumen	modified	modified
	(unaged)	bitumen	bitumen	(aged)	bitumen (aged)	bitumen (aged)
		(unaged)	(unaged)			
0	52			48		
2		52	59		58	58
4		62	65		67	65
6		67	72		73	74





The change in the penetration value and the softening point is from the increase in the additive content (Lignin 1 and Lignin 2) of varied percentages of 2%, 4% and 6%. The results obtained is tabulated in table 6 and 7 and plotted in the figure 5 and 6 The penetration value for the unaged modified bitumen decreases as of increase in the bitumen content, the same continues in the RTFOT aged modified bitumen. And makes the binder harden. As of decrease in the penetration value, there is drastically increase in the softening point of both similar result of RTFOT aged and unaged bitumen. The results for the both unaged and RTFOT aged bitumen the penetration value and softening value is almost similar.

# **Rotational Viscometer**

	Rotational viscometer					
Percentage bitumen %	Virgin bitumen (unaged)	Lignin 1 modified bitumen	Lignin2 modified bitumen	Virgin Bitumen (aged)	Lignin 1 modified bitumen (aged)	Lignin 2 modified bitumen (aged)
		(unageu)	(unageu)			
0	152			148		
2		160	168		154	160
4		212	240		200	224
6		324	318		302	290







The results of the rotational viscometer of unaged and RTFOT aged bitumen for 60/70 grade bitumen is shown in the table 8 and plotted in the figure 7 respectively. Here the results shows that the increase in the addition of the lignin content, than there will be increase in the viscosity of the binder. The results of the rotational viscometer is directly related to the penetration and the softening point tests results. The increase in the viscosity of the lignin modified binder is caused by the aging effect from heating during the processing and stiffening effect from the addition of the types of lignin.



#### **Aging Index**



The aging index is the ratio viscosity of the aged binder to the viscosity of the unaged binder. If the more is the aging index, more will be the degree of the aging of the binder. In the figure 8 the aging index is decreasing which indicates the reduction in the aging property of the modified binder hence can be seen that both lignin contents acts as an antioxidant to the binder content

# Marshall Properties of Conventional Mix

For the DBM conventional mix the Optimum Bitumen Content has to be find, the percentage of bitumen from 4.5% to 6.5% by weight of aggregate at an internal 0.5%. In the present study Marshall Method mix design is used and MORTH specifications are followed. The grade of bitumen used is 60/70. The results of test conducted to find the optimum bitumen content of the DBM mix are tabulated in table 9.

In present work, the optimum bitumen content is evaluated based on the test results in DBM mix Based on the Marshall stability, 4% of air voids and maximum bulk density the optimum bitumen content is calculated. From the Table maximum stability is obtained at 5% bitumen content. Bulk density is obtained at 5% of bitumen content. Air voids at the 4% at the 5% of bitumen content. Graphs are plotted for each characteristic. The OBC is 5% ((5+5+5)/3=5%) for DBM mix.

Percentage of bitumen (%)	4.5	5	5.5	6	6.5
Marshall stability (kN)	14.66	15.3	14.36	13.48	12.13
Flow value (mm)	2.8	3.3	3.6	3.7	4.1
Marshall quotient ( stability /	5.23	4.6	3.98	3.64	2.9
flow)					
Bulk density Gb (g/cc)	2.38	2.388	2.384	2.383	2.38
Air voids Vv (%)	4.94	4.06	3.574	2.939	2.34
Volume in mineral aggregates VMA	15.46	15.774	16.43	16.96	17.52
(%)					
Voids filled with bitumen VFB (%)	68	74.21	78.24	82.67	86.63

Table 9: Marshall Test results of the conventional mix



Figure 9: Bitumen content v/s Marshall Stability for the DBM mix with the Stone Dust as Filler Materials



Figure 10: Bitumen Content V/S Flow Value for the DBM Mix with the Stone Dust as Filler Materials

Figures 9 and 10 shows the Marshall stability and the Flow value for the DBM mix with the stone dust as filler materials. There is increase in the stability as of the bitumen content increases, 5% is the maximum stability and after 5% leads to decrease in the stability as on increase in the bitumen content. The maximum stability is 9kN as of MORTH specification 2013. The maximum stability at the 5% of bitumen content is 15.3kN. The flow value depends on the stability value. When there is increase in bitumen content there is increase in flow value due to the decrease in the stability value



Figure 11: Bitumen content v/s bulk density for the DBM mix with the stone dust as filler materials

From figure 11 shows the increase in the bulk density till 5% and further there is gradual decrease in the density due to the increase of the bitumen content. The bulk density is obtained is 2.388g/cc at 5%. Bulk density depends on the weight of sample in air and water which further depends on the compaction factor.



Figure 12: Bitumen content v/s Air voids for the DBM mix with the stone dust as filler materials

From figure 12, the air voids decreases when increase in the bitumen content. The maximum air voids is 3 to 5% as of the MORTH specification 2013. Bitumen content at 4% air voids is obtained at 5% and satisfies the MORTH specification 2013. Thus, 5% optimum binder content is considered based on the maximum stability, density and 4% air voids.



Figure 13: Bitumen content v/s voids in mineral aggregates for the DBM mix with the stone dust as filler materials

From the figure 13, if there in increase in the bitumen content there in increase in the VMA. This increase is due to enhance the mix properly of the DBM mix when increase in bitumen content. The VMA value varies form maximum is 14% as per MORTH 2013. The increase in the VMA from 4.5 to 6.5% indicates the low mixture property of stability.



Figure 14: Bitumen content v/s voids filled with bitumen for the DBM mix with the stone dust as filler materials

From figure 14 the VFB values increases with increase in the bitumen content. The VFB values obtained at 5% as 74.21. As per MORTH specification the maximum VFB is within 65 to 75%. Thus the VFB value satisfy the MORTH specification.

# Indirect Tensile Strength (ITS) and Tensile Stress Ratio (TSR)

#### Table 10: Indirect Tensile Strength Results for DBM Mix

Additive	%	ITS, conditioned (MPa)	ITS, unconditioned, (MPa)	%TSR (MPa)
Nil	0	0.218	0.347	62.82
Lignin 1	2	0.385	0.523	73.61
	4	0.744	0.709	93.11
	6	0.633	0.716	88.40
Lignin 2	2	0.460	0.570	80.70
	4	0.660	0.740	89.18
	6	0.740	0.854	86.65

The indirect tensile strength and tensile stress ratio results of the DBM mix with the additive of Lignin 1 and Lignin 2 of varying percentages for both conditioned and conditioned are tabulated in the table 10. As of the values of the indirect tensile strength and tensile stress ratio from the table, there was the improvement in the tensile strength compared to the conventional DBM mix with addition of the additives. The additive used here is the lignin 1 (organic lignin) and Lignin 2 (Lignosulphonates) at varied percentages



Figure 15: Lignin 1 Content V/S Indirect Tensile Strength for the DBM Mix



Figure 16: Lignin 2 Content V/S Indirect Tensile Strength for the DBM Mix

From figure 15 and 16, as of the increase in the lignin 1 and lignin 2 content as an additive there was greater increase in the indirect tensile strength value compared to the conventional mix of DBM. As increase in the indirect tensile strength in both conditional and unconditional up to 4% and further increase in the additive content to 6% there is decrease in the indirect tensile strength, this behaviour because the tensile strength is directly related to the function of the binder and its content for mix, stiffening effect and bonding property. If the additive content further increase in beyond limit, leads to loosen of the bonding property between the aggregates, binder and additive and even leads to the increase in the viscosity property of the binder which increase the volume of the additives used.



Figure 17: Variation of TSR% value from the conventional mix, lignin 1 and lignin 2

As of the Figure 17 the TSR values of conventional mix is 62.82% which is less than 80% minimum TSR values as per the MORTH 2013. The obtained TSR values of the modified mix with the additives of Lignin 1 and Lignin 2 is higher value then that is specified in MORTH. The maximum TSR value of lignin 1 is 93.11 and Lignin 2 is 89.19. From the figure 4.12 the variation between the minimum % TSR values can be observed.

The results of TSR which represent the moisture susceptibility increased up to some percentages and found later on decreasing, due to the addition of the additive content gives the TSR value than the conventional mix. It indicated the less in water sensitivity, less moisture content, increase in the resistance to crack, fatigue life and even the stability parameters.

#### **Fatigue Test**

A constant repeated load was applied at a loading frequency of 1Hz with a rest period of 09 sec. having type of loading waveform was used. The load repetition was continued till the specimen failed that is when the permanent horizontal deformation results is 3 mm. Fatigue life is the number of load

repetitions to cause failure of specimen. The specimens were tested at 27 <sup>o</sup>C temperature at varying stress levels. As tabulated in the below table 4.6 the results of the fatigue life cycle for the conventional mix, lignin 1 and lignin2. The stress level opted here is 10%. The load is taken of 2T.

From the table 11, the maximum no. of cycles is considered and plotted below in the figure 18 These no. of cycles gives the durability of pavements, its stability and t=fatigue life.

# Table 11: Fatigue Life Cycles

Stress Level	Fatigue test number of cycles				
	Conventional mix	Lignin 1	Lignin 2		
10%	1162	2365	2544		
10%	1196	1944	2314		
Average	1179	2154	2429		



# Figure 18: Fatigue Life Cycles

# FTIR Analysis



# Figure 19: spectrum analysis the plot between the Transmittance % (frequencies of light) v/s Wavenumber cm<sup>-1</sup> (measured absorption intensity)

The graphical representation shown here in figure 19 give the FTIR analysis of the lignin, additive used in the modification of the asphalt binder. The FTIR passes to several test and get passed so that it gives the accuracy of lignin characteristics and its usage in the graphical representation. The resistance to the sulfoxide and the carbonyl functional groups which helps in enhancing the anti-oxidation property to reducing aging indices in the asphalt binder after the RTFOT aging process is evaluate by these analysis.

# Conclusion

The study was carried out on the lignin as an additive which is taken in proportion of 2%, 4%, 6%. The following conclusions are obtained; physical test, the rotational viscometer, the Optimum Binder content for the DBM mix, ITS and TSR percentages and its fatigue life is evaluated, the FTIR analysis is optimised.

- The basic tests were conducted for aggregates and bitumen satisfies the IS code.
- The addition of Lignin 1(organic lignin) and Lignin 2 (lignosulfonate) to asphalt binder caused significant rheological changes depending upon the type of the binder and type of Lignin.
- The physical properties are executed for the laboratory, there was decrease in the penetration value and increase in the softening point due to the stiffening effect of lignin.
- The viscosity values by using rotational viscometer and aging indices, it can be concluded that Lignin shows some antioxidant activity when added to asphalt binder.
- If the lignin's content increase in further proportion there increases in the stiffening effect. As increase in stiffening effect it less resistance to crack at low temperature itself.
- Based on the Marshall stability the, air voids and bulk density, the Optimum Binder Content (OBC) of DBM mix is 5%.
- The indirect tensile strength and the Tensile strength ratio for the conditional and unconditional specimens is evaluated. The ITS value is obtained for both Lignin 1 and Lignin 2 is at 4%. The TSR value is the maximum value obtained is for lignin 1 is 93.11 MPa and lignin 2 is 89.18 MPa.
- For the above maximum TSR value obtained, the fatigue life is executed. The no. of life cycle is obtained. For conventional mix is 1196 no. of cycles, lignin 1 is 2365 no. of cycles and lignin 2 is 2544 no. 0f cycles.
- The FTIR spectroscopy show that the addition of lignin can help resist the formation of carbonyl functional groups in asphalt binder after the RTFO. This suggests that lignin could be used as an antioxidant modifier.

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