Asphalt mixture performance and testing

EVALUATION OF COLD MIX ASPHALTS AND ADDITIVES, APPLIED IN TURKEY, DEVELOPING OF THE SPECIFICATIONS THAT CONCERN MANUFACTURING, USAGE AND QUALITY CONTROL

Tuğba ÖZTÜRK, Savaş Nedim TUTAN, Muhammet KOMUT, Sina KİZİROĞLU
General Directorate Of Highways, (KGM), Turkey

Abstract

The choise of materials used in bituminous layer significantly affects road construction and usage that is service ability. Despite the improvements in construction techniques and material properties, because of increasing number of vehicles and environmental impacts, structural or non structural deterioration or local collapse and pits may occur on the highways. We can sort reason of occurring pits in generally due to wrong construction techniques, missing quality controls, not enough quality of materials used, excess moisture in the pavements granular layers, freeze dissolution effect, insufficiency of traffic and pavement layers. Generally, small amounts of hot mix asphalt (HMA) are needed for the repair of this type of deterioration that occurs locally. Production after deterioration may not be economically or not always technically applicable. It is also impossible to produce and store HMA before the deterioration. For this reason, it is preferred cold mix asphalt which allows production and pre-storable and can be used if needed. But in Turkey there is no standard or sufficient technical specification for the composition and properties of these materials and the performance expected from the mixtures except for user catalogs and specifications of the companies that produce or market the cold asphalt mix and additives. İn this study, it is aimed to determine components, definition of physical properties (bitumen, aggregate, additive properties, etc.), determination of storing lifetime, classification, performance measurements, technical and administrative specifications applicable to Turkey and specifications for implementation of cold asphalt mixtures that used for patch in HMA. For this reason, firstly, chemical analyzes of the cold patch additives were made and after this development of the application specification will be made and the necessary tests will be carried out in order to determine performances of the cold mix.

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1. INTRODUCTION

The most important factors for performance of the highway after the repair are the aggregate, bitumen and additives existing in the cold mix asphalt repair material which provides the opportunity of pot-hole repairs on highway pavements even during winter period. It is also known that waste oils are being used for production of cold pathing material additives. It will be instructive to determine the chemical characteristics of the additives and performance comparisons between additives used in the same type of mixes for determination of additive-performance relations of Cold Mix Asphalt Additives used in the country.

The asphalt mixture used as a pavement material is a mixture of fine and course aggregate with bituminous bonding agent having adhesive characteristic. Cold mixes are possible to prepare by adding mineral aggregates to emulsions or liquid asphalts; also, they are possible to prepare by using viscous oils named as flux-oil or oils named as cut-back additives.

The difference of flux-oils from cut-back asphalts is that volatile substances available in these oils are having high evaporation temperatures and therefore they do not evaporate at asphalt mixing temperatures such that they are not harmful for the environment. Flux-oil additives in cold asphalt mixes drops the viscosity of binder for more long periods of time.(1)

2. EXPECTED CHARACTERISTICS OF COLD PATCHING MATERIAL ADDITIVE

The performance of cold asphalt mixes are negatively affected by the shortages in expected characteristics of the mix in addition to the challenges raised from the application. The expected characteristics of cold patching mixes:

- Patching mix should be resistant against horizontal and vertical sliding after it is placed and compacted,
- Adhesion of the mix should be sufficient, especially it should provide the required bond over heavily dusty and unsatisfactorily cleaned patch surfaces,
- Should have a good resistance against water, should decrease and eliminate binder-aggregate stripping,
- The durability of asphalt in the patch hole decrease with time resulting ravelling under traffic loads. The cohesion between aggregate and mix should be sound in the patching mixture, should decrease the ravelling arising with time,
- There should be sufficient sliding resistance especially at long and wide patches,
- Patching mix should be soft enough and workable, there should not be agglomeration within the mixture,
- The patching mix should keep its workability after 6-12 months storage period,
- It should be resistant against freeze-thaw cycles. (2)

3. RESEARCHES

3.1. Determination of cold patching mix additives

Determination of characteristics of aggregate, bitumen and additive types in the content of Cold Patching Mixes that are used for repair of patches raised over highway pavement layers, are the most important points for the performance of the highway after repair. It is known that waste-oil is being used during production of Cold Patching Additives. Since there is not enough information rather than the declarations of Cold Patching Additive Manufacturers', the priority had given to the studies for determination of chemical characteristics of additives.

Within these studies, most commonly used Cold Patching Additives for the pavement layers have been coded 7 different groups as SYK1, SYK2, SYK3, SYK4, SYK5, SYK6 and SYK7.

3.2. Tests applied on cold patch additives

3.2.1. Cold patch additives heavy metal analysis

It is possible to determine if there exists waste-oil or not through heavy metal analysis of selected seven types of additives. Heavy metal analysis has been applied on SYK1, SYK2, SYK3, SYK4, SYK5, SYK6 and SYK7 additives and the results are given on Table.1.
The analysis of 22 elements (Fe, Pb, Cu, Cr, Al, Sn, Si, Na, Zn, Ag, P, Ca, Ba, Mg, Mo, Ti, V, B, Ni, As, Mn, Cd) have been realized in consequence with ASTM D 6595 method as to determine the amount of heavy metals as suspended and dissolved in the additive.

Table 1. Result of tests of determination of heavy metal amounts

	Fe	Cr	Pb	Cu	Sn	Al	Ni	Ag	Si	B	Na	Mg	Ca	Ba	P	Zn	Mo	Ti	V	Mn	Cd	As
SYK1	0,52	0,0	0,18	0,0	0,0	0,18	1,34	0,36	0,0	0,0	0,98	0,00	0,00	0,24	37,50	0,61	0,29	1,14	0,00	**3,11**	0,00	0,85
SYK2	45,7	0,0	0,0	0,5	1,04	1,19	1,34	0,38	0,0	0,0	**16,8**	0,56	0,00	0,22	106,5	0,16	0,65	1,52	0,00	**3,27**	0,00	1,34
SYK3	10,2	0,0	0,09	0,0	**12,1**	0,74	1,19	0,35	1,64	0,0	0,22	0,00	0,15	0,32	0,19	0,28	0,75	1,25	0,00	**3,25**	0,00	0,81
SYK4	0,41	0,23	0,0	0,2	**9,4**	0,12	0,0	0,0	0,87	0,1	0,0	0,00	0,00	0,05	0,00	0,26	0,81	0,02	0,49	0,15	0,18	0,00
SYK5	1,53	1,95	0,08	0,8	3,6	0,0	0,05	0,0	6,83	0,08	**6,62**	0,42	0,56	0,03	0,00	1,33	0,52	0,94	0,00	0,00	0,16	0,76
SYK6	126	< 1	0,0	1,4	46,3	12,0	0,0	0,0	< 1	< 1	32,0	< 1	< 1,1	0,00	33,4	< 1	< 1	< 1	0,0	0,0	< 1	0,0
SYK7	< 1	< 1	< 1	0,0	1,1	0,0	< 1	< 1	< 1	0,0	1,0	0,0	0,0	< 1	0,0	< 1	< 1	0,0	< 1	< 1	0,0	0,0

After inspection of Table 1., any distinct heavy metal pollution is not counted.

3.2.2. Kinematic viscosity

The kinematic Viscosity values of cold asphalt additives have been measured by using automatic viscosimeter device. For measurement, a 1.5 ml sample is injected in the viscosimeter column. The value read on the device is the kinematic viscosity value which is obtained by multiplying the device calibration constant with average flow time.

3.2.3. Determination of density

The densities of cold asphalt additives have been calculated by using U-tube principle. The samples are filled in the measurement cell of the device by means of the pipet-type pump available with the device or by means of injection. A temperature sensor at the cell of the device measures the sample temperature.

3.2.4. Total acid number

The acid number of additives have been determined by potentiometric titration. The sample is weight and resolved in resolvent (50% toluene+ 49% isopropanol alchol+1% water) and titrated in 0.1 M potassium hydroxide solution in isopropanol alcohol. The turning point of titration is taken as the last turning point of titration curve and the titration is finalized.

3.2.5. Determination of total water

Samples are being weight and injected in a titration pot of a device in which the iodine liberates coulometric at anode. While the water is fully titrated, the turning point that the iodine liberates is fixed by using electrometric turning point detector and the titration is ended.

3.2.6. Determination of refractive index

The refractive index of additives has been determined by refractometer. The device is used to determine the refractive index value at light having 589,3 mm wave-length.

3.2.7. Determination of flash point

Flash points of samples have been determined by flash point device by using 1- or 2-ml sample with closed cup method.

3.2.8. Ph measurement

pH measurement has been done by pH metre. Measurements have been realized directly without dilution.

The results of analysis for kinematic viscosity, density, acid number, total water, refractive index, flash point and pH values are given on Table 2.

Table 2. Cold asphalt additives analysis results

Analysis	SYK1	SYK2	SYK3	SYK4	SYK5	SYK6	SYK7
Kinematic Viscosity cst (mPa.s); 37,8 °C	37,5	8,79	5,11	2,64	4,81	8,23	1,498
Kinematic Viscosity cst (mPa.s); 40 °C	34	8,32	4,92	2,534	4,65	7,86	1,463
Kinematic Viscosity cst (mPa.s); 100 °C	5,52	2,75	1,79	-	2,07	49,0	30,0
Density (gr/cm^3); 15 °C	0,878	0,899	0,876	0,814	0,866	2,69	0,756
Density (gr/cm$^{3)}$; 15,6 °C	0,877	0,898	0,875	0,814	0,865	34,0	28,0
Density (gr/cm^3); 20 °C	0,875	0,895	0,873	0,811	0,862	0,894	0,805
Acid Number (mg KOH/gr)	-	0,024	0,772	0,095	1,365	0,893	0,804
Total Water (ppm)	2720,2	1078,9	1763,4	353,8	1933,5	0,891	0,802
Refractive Index (n)	1,49	1,46	1,47	1,45	1,46	11,93	0,022
Flash Point (Closed Cup) (°C)	200	186	122	111	88	2680,6	417,1
pH (25°C, direct)	8,35	3,6	6,4	4,36	9,05	1,45	1,45

3.2.9. Gas chromatography

Table 3. GC analysis results of vegetable based products

Oil Acid Combination	SYK2	SYK3	SYK5	SYK6
Palmitic acid	9,32	6,28	6,07	16,05
Stearic acid	3,14	3,45	4,64	7,65
Oleic acid	34,79	19,54	35,33	33,36
Linoleic acid	43,38	46,25	16,09	33,92
a-Linoleic Acid	4,93	2,2	4,23	1,44
Other	4,44	22,28	33,64	7,58

Change in oil acid shall be followed for shelf-lives of vegetable-based products on Table 3.

3.2.10. Distillation

Since the volatility which is the base of distillation is also a distinguishing parameter for cold asphalt additives, cold asphalt additives have been distilled by simple distillation method. For this purpose, to determine the distillation of liquid additives shall be done at atmospheric conditions or under vacuum, SYK3 has been selected randomly and the laboratory tests have been initialized.

Table 4. Distillate percentages obtained by vacuum distillation at different temperatures

Distillation Range (°C)	SYK1	SYK2	SYK3	SYK4	SYK5	SYK6	SYK7
100	-	-	-	-	10	-	-
120	-	-	-	-	-	-	-
140	-	-	-	20	-	-	-
160	-	-	5	66	-	-	-
180	2	-	-	-	-	-	-
200	4	-	14	68	20	-	-
220	21	-	19	70	24	-	-
225	60	-	-	-	-	-	-
240	-	20	30	94	36	-	-
250	-	-	42	-	84	-	-
260	-	68	61	-	-	-	-
265	-	-	85	-	-	-	-

From Table 4., 60-80% of SYK5, SYK3 and SYK2 additives can be distilled approximately at 250°C, SYK1 additive at vapour temperature of 225°C by volume. Sample SYK4 is composed of two different materials having evaporation temperatures around 150°C and 240°C.

3.2.11. FTIR structure lighting

FTIR spectrum device is used to define organic compounds. IR spectrum of all compounds, except optic isomers, are different from each other. IR zone is in between visible zone of electromagnetic spectrum and micro-wave zone. This zone is between 4000-450 cm^{-1} wave length. IR spectrum provides direct information for the structure of organic compound. However, do not provide information about any material if it is pure or not.

The horizontal axis in FTIR spectrum represents the wave length (cm-1), vertical axis represents % T.

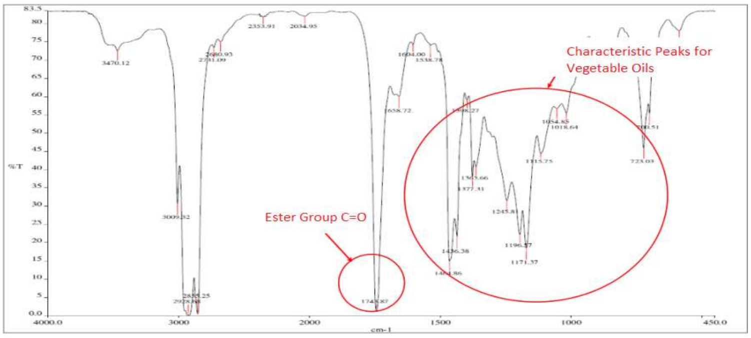

Figure 1: SKY5 FTIR spectrum

From figures, it is identified that SYK2, SYK3, SYK5 and SYK6 samples are vegetable oil based, other samples are chemical based

3.3. Gradation and characteristics of mix

The aggregate to be used in the mix has been supplied from Malıköy Quarry in Ankara City. The aggregate is lime-stone based. Tests have been done on course, fine and mineral filler aggregates to determine their characteristics at Directorate of Highways-Department of Research and Development Laboratories.

3.3.1. Sieve analysis

Gradation that is obtained at the end of the sieve analysis to find the grain size of aggregates is used to classify the aggregate groups, to check the compliance with the specification limits of specific layer that the aggregate is planned to be used and mix-design calculation of aggregate groups. The sieve analysis has been done in consequence with ASTM C-136 and TS EN 933-1.

Table 5. Sieve analysis test results

Sieve Size		Design Gradation	HTS Section 409 Type-2
mm	inch		
19	3/4"	100	100-100
12,5	1/2"	92,0	88-100
9,5	3/8"	82,0	72-90
4,75	No.4	46,0	42-52
2,00	No.10	29,0	25-35
0,425	No.40	13,0	10-20
0,18	No.80	9,0	7-14
0,075	No.200	4,5	3-8

The gradation used in this study has been selected from Highways Technical Specification-Section 409-Plant-Mixed Bituminous Maintenance Material-Table 409-1, Type-2. Since the pot-holes are mainly detected over wearing course layers, the selected Type-2 is equivalent to the wearing course mixture grain sizes.

3.3.2 Los Angeles fragmentation resistance determination test

The wearing resistance of aggregate is determined by Los Angeles test. The measurements have been done in consequence with TS EN 1097-2. The Los Angeles Fragmentation Value of Malıköy Quarry Aggregates is found to be 27%. The higher resulting braking down amount of aggregates under the effects of climate, traffic and both negatively effects both stability and durability of the mix. The aggregates from Malıköy Quarry is not expected to result any stability problem during the test studies considering that it complies with the Highways Technical Specification of General Directorate of Highways.

3.3.3 Magnesium sulphate ($MgSO_4$) freezing lost test

Test is applied on the aggregate 10 mm between 14 mm as 5 times freezing-thawing periods of 48 hours duration and the percentage of loss at the end of these 5 periods is calculated. During the test Magnesium Sulphate (MgSO4) solution is used. Measurements have been done in consequence with BS 812:105. The freezing-thawing test results applied on Malıköy Quarry Stone which complies with the Highways Technical Specification of General Directorate of Highways. Since the wearing course layer is the most effected layer from freezing-thawing cycle and the cold asphalt mixes shall be used on wearing courses, any degradation or durability problem is not expected according to this test result.

3.3.4 Determination of flakiness index

Any asphalt layer composed of flat aggregates is not stable under the traffic load. The flat grains are broken easily under the load and decreases the strength of the mix. Therefore, knowing the flakiness ratios of aggregates have importance regarding the adhesion of bitumen to aggregate surface and interlock of aggregates with each other. Measurements have been realized in consequence with TS EN 933-3. The flakiness index of Malıköy Quarry Stone, which has been used during the tests, indicates that it is cubical.

3.3.5 Methylene blue test

Measurements have been realized according to TS EN 933-9 during Methylene Blue Test. Since the clay amount achieved during Methylene Blue Test for Malıköy Quarry Stone is low enough to comply with Highways Technical Specification of General Directorate of Highways criteria, it is not expected to face with swelling and low stability problems during the tests.

3.3.6 Specific weight and absorption test

The specific weight of aggregate is the ratio of density of aggregate to the weight of same volume of water at 25°C. For air voids calculation of compacted pavement, the maximum theoretical specific weight of aggregate is calculated considering the amount of bitumen amount absorbed by the aggregate. Measurements have been realized according to TS EN 1097-6. The water absorption value obtained from the tests show that the sensitivity of Malıköy Quarry Stone against water is low.

3.3.7 Peeling resistance test

The loss of adhesion between aggregate and bitumen under the effect of water and temperature is determined by the Peeling Resistance Test. The amount of bitumen peeling is dependent on the type of aggregate (limestone, basalt, etc.) and type of bitumen binder. Measurements have been realized according to TS EN 12697-11.

Figure 2: Peeling resistance test sample from Malıköy Stone Quarry

Based on the results obtained from tests on Malıköy Quarry Stone it is inspected that the stone has high peeling resistance according to HTS criteria and the bond between aggregate and bitumen is high, any anti-stripping additive has been used.

Table 6. Aggregate test results

Name of Test	Result of Test	HTS Part 409-2	Test Standard
Los Angeles Wearing Lost, %	27	≤ 30	TS EN 1072-2
$MgSO_4$ Frost Lost, %	4	≤ 18	TS EN 1367-2
Flakiness Index, %	19	≤ 25	TS EN 933-3
Methylene Blue	0,75	≤ 2,0	TS EN 933-9
Water Absorption, %	0,9	≤ 2,5	TS EN 1097-6
Peeling Resistance	65-70	≥ 50	TS EN 12697-11

The aggregate test results on Table 6. reflects that the aggregate characteristics are complying with the Highways Technical Specification Part 409 to be used as cold pathing material.

3.4 Bituminous binders

B70/100 penetration bituminous binder supplied from Kırıkkale Refinery has been used.

3.4.1 Penetration test

The test sample is heated up to estimated softening point (TS EN 1427) of 100 ºC and filled in to a clean (free from dust, oil, rust, etc.) test sample cab. After cooling the sample to determination temperature, the sample is filled in to the test cab such that the needle is 10 mm to estimated penetration depth. Measurements have been realized according to TS EN 1426.

3.4.2 Softening point test

In order to measure the sensitivity of bituminous binder against temperature (the temperature that the bitumen is starting to flow) the temperature named as softening point is determined by ring-ball method. Since the softening point of bitumen's with very high viscosity is high, the production and working temperatures of hot-bituminous mixes should also be high. Measurements have been realized according to TS EN 1427.

3.4.3 Flash point test

Bituminous materials release volatile compounds at higher temperatures based on the degrees. These volatile compounds burn and results flaring. This situation is very dangerous and it is necessary to determine this temperature for each bitumen. Measurements have been realized according to TS EN 2592-Cleveland Open Cab Method.

3.4.4 Specific weight test

Density (ρ): Is the mass of unit volume of a material. The unit is kg/m3. The standard temperature to determine the density is (25,0 ± 0,2) ºC. Specific weight is determined by pycnometer in general. Measurements have been realized according to TS EN 15326.

3.4.5 Brookfield Viscosity Test

Viscosity is a scale related with the resistance to flow and consistency of a bituminous binder. While the consistency increases, that means the bitumen is approaches towards the solid state, viscosity value increases. The purpose of the viscosity test is to determine the flow characteristics of bituminous binders at working temperatures during applications. Measurements have been realized according to ASTM D 4402.


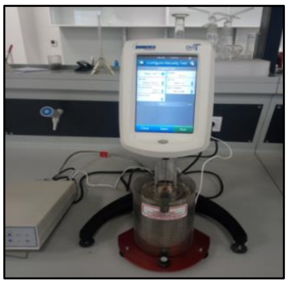

Figure 3: Brookfield test

Table 7. B70/100 bitumen test results

Name of Test	Result of Test	HTS Part 412-1	Test Standard
Penetration 25 °C, 0,1 mm	70	70-100	TS EN 1426
Softening Point, °C	48,2	43-51	TS EN 1427
Flash Point, °C	330	≥230	TS EN ISO 2592
Specific Weight, g/cm^3	1,028	1,0-1,1	TS EN 15326
Brookfield Viscosity, 110 °C, cp	1590	-	ASTM D 4402
Brookfield Viscosity, 135 °C, cp	192,5	-	ASTM D 4402

B70/100 Kırıkkale Bitumen test results on Table 7. reflects that the bitumen characteristics are complying with the Highways Technical Specification Part 409. Penetration, Softening Point, Loss in Mass and Brookfield Viscosity values of B70/100 Kırıkkale Bitumen have been measured by mixing the cold patch additives.

3.5 Bituminous binder and cold patch additive mixture

It is proposed to test penetration and softening point values of B70/100 bitumen by mixing with 6 different cold patching additives, but the tests have not been realised due to the reason that the consistency of samples were very soft.

3.5.1 Loss in mass test

Due to ageing and release of volatile gasses within the bitumen with time, some amount of loss in mass is inevitable. But, for some bitumen, due to formation of oxidised compounds an increase in mass can also be happen. Loss in mass test have been done for each additive mixed with B70/100 bitumen and it has been observed that the highest amount of loss in mass is on SYK4+B70/100 sample. In addition, according to the results of vacuum distillation on Table 2.4, SYK4 has an easy evaporation characteristic at low temperatures resulting high amount of loss in mass. Measurements have been realized according to TS EN 12607-1.

Table 8. B70/100+additives loss in mass test results

Additives	Empty Beaker Weight (gr)	Sample + Empty Beaker Weight (gr)	Waited Sample + Empty Beaker Weight (gr)	Loss (%) (24 hours 25 °C)
SYK1	51,549	115,499	115,473	0,04
SYK2	51,953	110,42	110,331	0,15
SYK3	49,175	102,324	101,027	2,44
SYK4	52,525	89,375	86,953	6,57
SYK5	52,208	102,34	100,025	4,61
SYK6	49,010	105,196	104,928	0,48

3.5.2 Brookfield viscosity test

Table 9. B70/100+ SYK1 sample Brookfield viscosity test results

15% SYK1 + B70/100		20% SYK1 + B70/100		25% SYK1 + B70/100	
Temperature °C	Viscosity cp	Temperature °C	Viscosity cp	Temperature °C	Viscosity cp
90	1388	80	1870	70	2498
100	829	100	752	110	207,5
				120	135

Table 10. B70/100+ SYK2 sample Brookfield viscosity test results

15% SYK2 + B70/100		20% SYK2 + B70/100		25% SYK2 + B70/100	
Temperature °C	**Viscosity cp**	**Temperature °C**	**Viscosity cp**	**Temperature °C**	**Viscosity cp**
60	2406	70	1320	60	1710
70	1847	100	230	70	915
80	1288	110	152.5	80	592

Table 11. B70/100+ SYK3 sample Brookfield viscosity test results

15% SYK3 + B70/100		20% SYK3 + B70/100		25% SYK3 + B70/100	
Temperature °C	**Viscosity cp**	**Temperature °C**	**Viscosity cp**	**Temperature °C**	**Viscosity cp**
70	2333	60	2243	50	1673
110	217,5	70	1720	60	797,5
120	137,5	100	240	70	238,5
		110	147,5		

Table 12. B70/100+ SYK4 sample Brookfield viscosity test results

15% SYK4 + B70/100		20% SYK4 + B70/100		25% SYK4 + B70/100	
Temperature °C	**Viscosity cp**	**Temperature °C**	**Viscosity cp**	**Temperature °C**	**Viscosity cp**
70	1475	60	1809	60	1503
110	150	70	1250	70	944

Table 13. B70/100+ SYK5 sample Brookfield viscosity test results

15% SYK5 + B70/100		20% SYK5 + B70/100		25% SYK5 + B70/100	
Temperature °C	**Viscosity cp**	**Temperature °C**	**Viscosity cp**	**Temperature °C**	**Viscosity cp**
80	1495	60	1763	60	1780
100	377	70	1068	100	137,5

Table 14. B70/100+ SYK6 sample Brookfield viscosity test results

15% SYK6 + B70/100		20% SYK6 + B70/100		25% SYK6 + B70/100	
Temperature °C	**Viscosity cp**	**Temperature °C**	**Viscosity cp**	**Temperature °C**	**Viscosity cp**
50	2698	50	2369	50	2228
60	2139	60	1810	60	1128
70	1580	70	1251	100	137,5

The Brookfield viscosity test results of 6 different cold patch additive and 70/100 bitumen mixtures provide us to determine the mixing ratios and mixing temperatures of selected additive types within this study. Additives are added to B70/100 bitumen at 135 ºC and the temperatures have been dropped so that the workability values have been achieved.

3.6 Preparation of bituminous mix

Practical density, void ratio, voids between aggregates values have been calculated on the maintenance material mixes prepared by using cold asphalt additives and the hot prepared maintenance material mixes and maximum theoretical specific weight, stability, conditional stability, indirect tensile strength test and workability tests are applied on these mixes.

3.6.1 Bituminous mix design by Marshall method

Marshall method can only be applied on hot mixed asphalt pavements by using aggregates with maximum grain size of 25 mm (1 inch) or less and bitumen with known penetration and viscosity characteristics. This method is used. in our Country, for design of bituminous pavements. The procedure for Marshall Method starts with the preparation of test sample. Design form for Bituminous Hot Mix is given as enclosure.

Figure 4: Preparation of marshal test sample by SYK3 additive

Table 15. Marshall test results

Name of Test	**SYK1 % 25**	**SYK2 % 20**	**SYK3 %20**	**SYK5 % 25**	**SYK6 % 25**	**Hot Design**
Practical density compaction (145 ºC)	-	-	-	-	-	2,407
Practical density compaction (80-90 ºC)	2,414	2,415	2,414	2,412	2,404	-
Practical density cold compaction (25ºC)	2,242	2,300	2,287	2,255	2,254	-
Maximum theoretical specific weight	2,509	2,594	2,508	2,519	2,588	2,517
Air void mixing (80-90 ºC)	3,8	6,9	3,7	4,2	7,1	4,4
Air void cold mixing (25ºC)	10,6	11,3	8,8	10,5	12,9	-
VMA mixing (80-90 ºC)	14,3	14,2	14,3	14,3	14,6	14,5
VMA cold mixing (25ºC)	20,4	18,3	18,8	19,9	19,9	-

3.6.2 Marshall stability and flow test

The total load amount which results the sample to be broken is named as “Marshall Stability”.

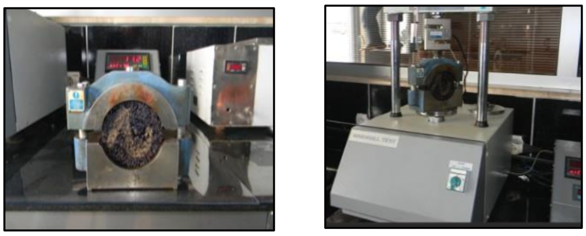

Figure 5: Stability test on sample prepared by SKY3 additive

Table 16. Stability test results

Name of Test	SYK1 % 25	SYK2 % 20	SYK3 %20	SYK5 % 25	SYK6 % 25	Hot Design
Normal Marshall stability, kg (40 min. at 25 ^{0}C', 60 ^{0}C' water bath) hot compaction	980,0	489,0	709,0	762,0	584,0	1153,0
Conditional Marshall stability, kg (96 hours at 25 ^{0}C', 72 hours at 60 ^{0}C' water bath) hot compaction	885,0	561,0	771,0	782,0	650,0	1144,0
Stability ratio, %	90,3	114,7	108,7	102,6	111,3	99,2

3.6.3 Indirect tensile strength test

Indirect Tensile Strength test is applied to determine the strength of the mix against freezing-thawing effects and harmful effects of water.(TS EN 12697-12, AASHTO T 283)

Table 17. ITS test results

Name of Test	SYK1 %25	SYK2 %20	SYK3 %20	SYK5 %25	SYK6 %25	Hot Design
Normal ITS, kg/cm^2 (2 hours at 25 ^{0}C' water bath) hot or cold compaction	Could not be realized	Could not be realized	Could not be realized	Could not be realized	Could not be realized	9,90
Conditional ITS, kg/cm^2 (%70-%80 saturation, 16 hours at -18 ^{0}C deep freeze, 24 hours at 60 ^{0}C' water bath, 2 hours at 25 ^{0}C' water bath) hot mix	-	-	-	-	-	8,68
Conditional ITS, kg/cm^2 (10 min. Vacuum in water, 30 min. İn water without vacuum, if extension in volume $\leq$ %2, 72 hours 25 ^{0}C water bath) cold compaction	Could not be realized	Could not be realized	Could not be realized	Could not be realized	Could not be realized	
ITS ratio, %	-	-	-	-	-	87,7

3.6.4 Workability test

The performances of pit patching materials have been inspected by Texas Department of Transportation and the Federal Highway. Both cut-back and emulsion mixes have been studied in lab and at site on the mixes supplied from different companies.(2) In order to compare the performances of different cold patching materials, all cold mixes have been kept in oven to make them stable. In the light of this study, a method has been developed by the General Directorate of Highways and is explained below.

The bituminous asphalt maintenance mixture is pre-mixed and its temperature is brought to ambient temperature. A pvc sample cab is prepared and the inside surface is oiled by using a selected material which is known that it shall not create harmful effects to the mixture. The pre-prepared mixture is filled inside the pvc sample cab as two layers such that the final height shall be 20 cm. Compaction is applied by keeping Marshall hammer 10 seconds freely on the sample and then 10 times dropping the hammer from highest level.

In this way, the top cover of the sample preparation mold, which is compressed into two layers of mixture sample, is also tightly closed and subjected to conditioning at 40 C for 24 hours vertically. After the conditioning process is complete, the compressed sample is removed from the mold with a suitable sample extractor in the laboratory at an ambient temperature of 25 ºC without deterioration. The sample of the mixture, which has been removed from the mold, is immediately placed in the middle of the wood spreading plate and the stopwatch is activated. Elapsed time of cylindrical compacted sample mixture which is initially 20 cm high and unable to bear its own weight due to the progressive increasing temperature effect, the upper part of the cylindrical shape to completely overturn or to collapse the sample mixture in the vertical direction and then overthrow, the mixture begins to slip over each other is measured. During this time, the sample temperature is measured every 5 minutes and recorded.

The final agglomeration time and agglomeration temperature of the sample is recorded. The stacked sample is immediately spread by hand with a spatula into the spreading mold until a uniform height is obtained and no agglomerated material remains. The spreading time is recorded by measuring with the stopwatch from the time the stacked sample is interfered with the spatula until the spreading process is completed.

Figure 6: Workability test on SYK3 sample

Table 18. Workability test results

Name of Test	**SYK1 % 25**	**SYK2 % 20**	**SYK3 %20**	**SYK5 % 25**	**SYK6 % 25**
Workability settlement time, s	2220	8700	3660	1440	2100
Workability placement time in mould, s	180	145	120	130	120

4. RESULT AND DISCUSSIONS

To define the physical characteristics, to classify and to measure the performances, tests have been realized on additives of cold bituminous asphalt pathing materials which are being used on bituminous asphalt pavements for patching purposes and the following results have been achieved.

- Chromatogram results Show that distillation results of additives and distillation products are similar. Since the distillation is done under vacuum and the distillates arriving with different temperatures could not be picked up separately, this is an expected result. It is also visualised that the results of gas chromatography analysis are compatible with the distillation results.

- From the results given on Table 1., it is inspected that the values for phosphor (P) (maximum 10) is not complying with the waste mineral oil category parameters and limit values. In case waste mineral oils are used for preparation of cold asphalt patching materials, it is interpreted that this is due to the phosphorous compounds that are being added to mineral oils during their preparation.

- FITR analysis has been applied to determine the chemical composition of cold patching additives. From the results of FITR analysis, it is determined that SYK2, SYK3, SYK5 and SYK6 samples are vegetable oil based, and the other samples are chemical based; and for more detailed definition of structures, samples are analysed by Gas Chromatography (GC) device. Oil acid determination studies have been realized on vegetable oil-based products and it is evaluated that the differences in composition of products are due to the difference of sources of oils used for production.

- Evaluation of distillation results according to HTS 2013, it is inspected that the sample SYK4 is not complying with the required conditions at 260-360 °C range. According to America Queensland MRST19 technical standard, minimum 80% of oils are required to be picked up under 350 °C for atmospheric distillation.

- Viscosity measurement results at 40°C show that all of the SYK1, SYK2, SYK3 and SYK6 (Table.2) samples are above the limit value (MTRS 19,2009). However, SYK1 sample is measured as 36.80 mPa.s which is 7 times above the limit value of 4.6 mPa.s. SYK1 sample resulted much higher value than the acceptable limit.

- For flash point values, the samples are above the limit value of 61.5°C (MTRS 19, 2009) and are acceptable. However, sample SYK7 has start burning without flashing, a reliable measurement has not been obtained (it shows flammable material characteristic). Regarding total water content, all of the SYK1, SYK2, SYK3, SYK4, SYK5, SYK6 and SYK7 samples are above the limit value 0.1% given on Table.2.

- Although they are not covered by Table 2. as the parameters for definition of cold patching material additives, considering the probability that they may be distinguishing characteristics, Flakiness Index and pH; acid number analysis for the possibility of use of waste mineral oil have been realized. It has been recorded that the flakiness index values of samples change between 1.45-1.49 and similar; the pH values are highly fluctuating. The pH values reflect that the additives some of the samples are acidic and some are basic.

- It is established from GC analysis results of chemical based products that there exists heavy alkyl benzene (HAB) and phthalate (probably DOP derivatives with molecular weight Mw: 390) in SYK4 sample; there exists heavy alkyl benzene (HAB) in sample SYK1 and there exists petroleum fraction between C10-C13 hydrocarbon range in SYK7.

- From loss on mass test results on Table 8., it is inspected that the values for samples SYK3, SYK4 and SYK5 are high and above the limit values. The loss on mass value 6.57% for SYK4 is due to its content of early evaporating chemicals. It is evaluated that the high amounts of loss on mass shall increase the number of voids in the mix resulting the stability problems.

- From Table 15. Marshall test results, it is inspected that 7.1% of air-voids value for hot-mix of SYK6 25% sample is higher relative to all other samples. This fact is resulting the decrease in the stability of SYK6 25% mix.

- Table 17. Indirect tensile strength tests have not resulted satisfactorily for all samples. Most of the samples have lost their strength after the conditioning and broken, no readings have been obtained from the device. It is evaluated that all samples are not durable against the harmful effects of water.

- As shown on Table 16., stability test results for all samples are high. The stability of SYK1 25% mix is higher than the other samples and stability after long-term conditioning is lower than the stability after short-term conditioning. However, the stability values of all other samples are increasing after long-term conditioning. This situation is thought to be due to the curative effect of the chemicals available in the additives on stability with time.

- Workability test results on Table 18. presents that the workability characteristic of SYK1 25% sample is low. This fact explains the highest stability value recorded on Table 16. for SYK1 25% sample. The low workability of SYK1 25% additive positively effects the stability. However, low workability may result to create laying and compaction problems during the applications.

- This study is being progressing at General Directorate of Highways-Research and Development Division to develop the application specification for cold patching additives.

- The performance of the bituminous mixture produced by using cold asphalt patch additives is lower than the bituminous hot mixture as well as it is considered that it is better to apply bituminous hot mixture instead of cold asphalt patch additives because of the harmful effects of water after the application and the seasonal effects.

5. REFERENCES

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