Chemically Stabilized Rubber Bitumen

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ABSTRACT

The production of rubber bitumen by blending of bitumen with crumb rubber from scrap tires is not just a waste treating method, but a possible solution for improving the durability of asphalt pavements compared to roads made of conventional bitumen. The following advantages are usually emphasized: improved durability, longer life time, lower life cycle / maintenance cost, noise reduction effect, etc.
The application of rubber bitumen is widely spread in the USA, where the production of the rubber bitumen is generally in situ, close to the asphalt mixing plant.
MOL Hungarian Oil and Gas Plc. together with University of Pannonia developed a new, patented production method (WO/2007/068990), which is capable to produce the Chemically Stabilized Rubber Bitumen (CSRB) in an oil refinery bitumen modifier units.
The effect of chemical composition of crumb rubber on CSRB properties was investigated. Its quality requirement was elaborated for suitable quality of CSRB.
CSRB can be transported, stored and applied as the generally used conventional modified bitumens. The unfavourable high viscosity of the classic rubber bitumen can be reduced to the level of the general produced polymer modified bitumen binders. Settling tendency of undissolved particles is dramatically reduced as well.
Asphalt tests have proved that the performance of CSRB is comparable to high quality polymer modified bitumen. Experiences of paving tests using CSRB attract the attention to the fact that the appearance of a binder of such quality is required in the future; it significantly supports the sustainability.

Keywords: rubber bitumen, chemically stabilized rubber bitumen, CSRB, crumb rubber, asphalt

1. INTRODUCTION

In industrially advanced countries 9 kg of waste tyres are generated yearly counting to a single person [1]. In the United States 2-3 billion illegally deposed scrap tyres exist, while the estimate for these historic stockpiles in the European Union stands at 5.5 million tons [2]. In Hungary 2.7 million cars are running, and yearly 40-50000 tons of scrap tyres are generated [3]. Producing crumb rubbers and blend with bitumens for paving purposes is a possible utilization method of scrap tyres.

Since the beginning of crumb rubber application for road construction and maintenance two groups of processes have developed: the dry and the wet process [4, 5]. The dry processes use the technology of adding coarse particles of crumb rubber directly into asphalt mix during bitumen and aggregate mixing [4, 5, 6]. During the wet process the crumb rubber is reacted with bitumen at elevated temperature and certain duration to obtain improved engineering properties [4, 5]. What occurs during wet process is the swelling and partial dissolution of the rubber but, mainly, absorption of the oils from bitumen into rubber matrix [5, 7]. The next step of wet process produces chemically modified rubber bitumens applying activation method of crumb rubber surface or using special compatibility improver additive [8, 9, 10]. These binders are more stable and the crumb rubber acts as an active modification agent in the rubber-bitumen disperse system.

The application of rubber bitumen is most widely spread in the USA, where the production of the rubber modified binder is in situ, close to the road construction. Normally the rubber bitumen binder needs to be mixed with aggregates within a few hours because of it has ability for phase separation [11]. The benefits of rubber bitumens and pavements constructed by their use in Europe are increasingly recognized. For example, in Portugal, in a government order of 2007 recognized the environmental and quality advantages promoted certification and acceptance of the product [12].

After the first trials it was quickly proved that the producing rubber bitumen means much more than just a utilization method of waste tyres. By using rubber bitumen in pavements it was proved that the quality and durability was enhanced compared to roads built with regular bitumen binders [4, 13]. The following advantages are usually emphasized regarding the rubber bitumen pavements: longer life, lower life cycle cost, wider utilization temperature range, noise reduction effect, less deformation and improved skid resistance, [4, 14, 15, 16, 17, 18]. Rubber bitumen production with suitable parameters of wet process can reach, in some properties even better values (e.g. cold cracking properties) than that of polymer modified bitumen [19].

Beside the benefits difficulties have reported in connection with rubber bitumen production and application, like high initial cost of rubber bitumen producing unit, recyclability, environmental concerns through emission, modification necessity of hot mix asphalt plant, sometimes difficulty in asphalt compaction [15, 20, 21, 22]. From the product quality point of view the limitations of rubber bitumen application and reasons of slow spread of this type of binder are as follows:

- application issues due to the generally high viscosity [15]
- unsuitable storage stability (quick phase separation) [22]
- impermanent product quality due to the altering quality of crumb rubber [23, 24].

A new production method, called ’improved wet process’, developed by Hungarian Oil and Gas Plc together with University of Pannonia [25]. Applying the new process the so called Chemically Stabilized Rubber Bitumen (CSRB) can be produced. The elaboration of the crumb rubber quality requirement and the application of ‘improved wet process’, it is possible to eliminate or to mitigate significantly the above mentioned issues.

The main objective of this work was to present the effect of composition of crumb rubber on the rubber bitumen quality. Test road construction using of CSRB was also evaluated. Performance comparison of CSRB to the polymer modified bitumen and 50/70 penetration grade bitumen is also presented.

2. EXPERIMENTAL SECTION

2.1 Materials

The bitumens used in this study were the 70/100 penetration grade with a softening point (ring and ball) of 46 °C and the 50/70 penetration grade with a softening point (ring and ball) of 51 °C were produced from Russian Export Blend crude oil by air-blowing of the vacuum residue.

For the experiments a wide range of crumb rubbers was applied, derived from passenger car tyres, truck tyres and technical waste rubbers, grounded at ambient temperature. The effect of crumb rubber particles’ size could be eliminated by using of crumb rubbers with nearly the same size distribution between 0-1.5 mm. The mean particle size of the particles, determined by screening, was between 0.5-1.0 mm. Their amount were 75 wt%.

The rubber analysis was completed according to ASTM-D297-93 standard [26]. Adherent water content, ash content, acetone and chloroform extract, polyisoprene content, total rubber polymer content and carbon black content were determined (Table 1).

A multifunctional additive was developed and used for improving the bitumen-rubber compatibility and the storage stability [14]. During the process the additive acted as a network forming material and improved the stability in this way. Viscosity increasing due to crumb rubber swelling process in the hot bitumen is well known phenomenon [7]. Viscosity decreasing effect of the additive is highlighted because of pumpability and applicability of the end product.

This agent has a surface active effect which contributes dispersing and dissolving process of agglomerated rubber particles in bitumen at the same time.

2.2 Methods and analysis

The improved wet process is able to produce the Chemically Stabilized Rubber Bitumen (CSRB) [25]. Blends of bitumen and crumb rubber were prepared by mechanical stirring in a high-shear batch labour mixer using a Silverson L4R agitator equipped with Square Hole High Shear Screen. Firstly multifunctional additive was poured than crumb rubber particles were slowly added at the initial stages of the mixing process and samples were processed for 60 minutes at 220 °C with a rotational speed of 400 rpm. The next step was cooling to 200°C and than additional mixing was used for 30 minutes with the same rotational speed. After the material was mixed, the resulting binder was poured into a small can and then investigated.
The composition of 23 CSRB samples produced with the use of 23 different crumb rubbers were follows:

- 60.0 wt% 70/100 penetration grade bitumen,
- 24.7 wt% 50/70 penetration grade bitumen,
- 15.0 wt% crumb rubber and
- 0.3 wt% multifunctional additive.

Table 1: Composition of crumb rubbers, CR-01 – CR-23

Crumb rubber	Composition, wt%							Compati-bility factor
	Adherent water content	Aceton extract	Chloro-form extract	Ash Content	Polyi-soprene content	Carbon black content	Total rubber hydrocarbon	
CR-01	0.6	10.8	1.1	7.6	26.7	28.1	51.8	1.45
CR-02	0.7	18.2	0.2	11.4	19.4	22.9	46.6	0.66
CR-03	0.5	11.7	0.2	5.1	19.0	32.3	50.2	1.13
CR-04	0.3	11.5	0.7	7.7	24.8	27.0	52.8	1.29
CR-05	0.6	20.9	0.5	10.5	9.1	30.0	37.5	0.29
CR-06	0.6	22.9	1.1	12.5	12.4	32.4	30.5	0.35
CR-07	0.7	26.1	0.8	15.3	6.7	23.4	33.7	0.16
CR-08	0.4	14.0	0.2	14.0	11.6	22.7	48.7	0.41
CR-09	0.6	6.6	0.1	5.0	44.1	27.8	59.9	3.80
CR-10	0.7	7.7	0.1	5.4	31.4	26.1	60.0	2.40
CR-11	0.2	8.1	1.2	7.1	29.8	27.3	56.1	1.96
CR-12	0.5	8.9	0.9	6.8	31.7	27.7	55.2	2.02
CR-13	0.3	10.7	0.3	6.1	26.8	28.3	54.3	1.60
CR-14	0.5	9.1	0.1	3.3	36.1	29.1	57.9	2.91
CR-15	0.3	7.1	0.1	4.1	42.8	31.4	57.0	3.82
CR-16	0.5	8.3	0.2	5.1	33.7	29.7	56.2	2.51
CR-17	0.4	7.6	0.2	5.4	30.0	28.1	58.3	2.31
CR-18	0.6	5.6	0.1	7.4	42.7	26.4	59.9	3.28
CR-19	0.3	8.9	0.1	8.4	31.5	27.3	55.0	1.82
CR-20	0.4	8.8	1.3	6.5	26.0	27.1	55.9	1.70
CR-21	0.4	8.9	0.2	11.4	31.5	23.9	55.2	1.55
CR-22	0.3	11.5	1.2	4.7	29.4	24.2	58.1	1.81
CR-23	0.3	7.4	0.6	6.8	34.6	26.7	58.2	2.44

Softening point, dynamic viscosity at 135 and 180°C were measured according to related standards like as EN 1427 and EN 13702-2. Storage stabilities of CSRBs have a great importance because of the intensive settling tendency of undissolved particles is one of the main problems of the conventional rubber bitumens. A tube with 2.5 cm diameter and 20 cm height was filled with CSRB sample, and then it was stored vertically at 180°C for 72 hours in an oven according to EN13399 standard. Then this tube was cooled in a refrigerator at about -20°C. Subsequently, the tube was cut into three equal sections. The samples taken from the top and bottom of the tube were used to evaluate storage stability by the measurement of the softening points. If the difference between upper and lower part is less than 5.0°C, the sample can be regarded as having good storage stability according to modified bitumen requirements in Hungary.

3. THE EFFECT OF CRUMB RUBBER COMPOSITION ON CSRB

Based on the data it was found that beside the technological parameters and the application of multifunctional additive for the production of CSRB a great importance is attributed to the quality of used crumb rubber. According to our laboratorial experiments a notable difference in the quality of rubber bitumens produced in the same conditions can be noticed even if the size distributions of crumb rubbers were the same. In certain cases intensive phase separation has been experienced due to settling rubber particles and any other undissolved particles derived from crumb rubber. In these cases softening point's differences above 10°C were experienced between the top and bottom of the samples. In other cases, however, only 1-2°C differences in softening point could be measured. Therefore crumb rubbers (Table 1.) were investigated in detail.

A strong relationship was observed between the investigated properties of the CSRBs and chemical composition of crumb rubbers. Linear or exponential correlations were found between their acetone extract, ash and polyisoprene content determined by ASTM D 297-93 method and above mentioned properties of CSRBs.

It was experienced that the acetone extract and the ash content had a reverse effect on the properties of CSRBs to the polyisoprene content. The effect of these rubber components have been also investigated together, therefore the so called compatibility factor (c_f) has been defined as follows:

$$c_f = \frac{polyisoprene\ content}{acetone extract + ash content}.$$

Compatibility factors of crumb rubbers are shown in Table 1. According to the measurements the higher value of compatibility factor resulted better storage stability of CSRBs which shown by lower difference in softening points (Figure 1a). Exponential relationship was found between storage stability and compatibility factor which correlation coefficient was 0.96. Between the softening point and the compatibility factor a linear correlation was observed (Figure 1b).

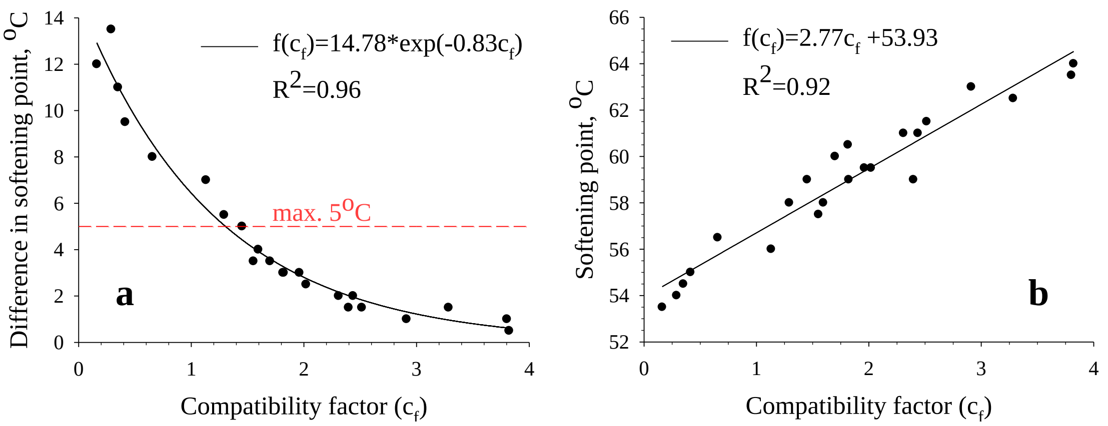

Figure 1 a-b: Effect of compatibility factor on storage stability (a) and softening point (b).

Similarly, higher compatibility factor (in other words higher polyisoprene content and smaller acetone extract and ash content) resulted higher viscosity (Figure 2 a-b).

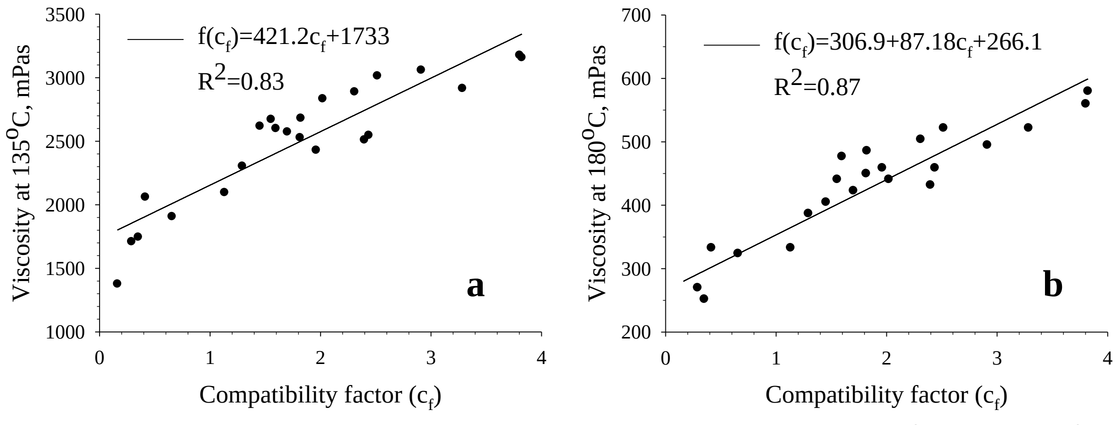

Figure 2 a-b: Effect of compatibility factor on dynamic viscosity measured at 135°C (a) and 180°C (b).

Suitable storage stability can be achieved without multifunctional additive using only crumb rubbers having the two highest value of compatibility factor (CR-09 and CR-15). Although difference in softening points during storage

stability investigation achieved the limit value; the measured difference between top and bottom was 5°C. It can be stated that storage stability was not adequate by using crumb rubber having compatibility factor less than 1.5 (crumb rubber samples of CR-01, CR-02, CR-03, CR-04, CR-05, CR-06, CR-07, CR-08). In these cases stability can not be improved significantly even if using multifunctional additive in higher content up to 2 wt%. These CSRBs contained so high content of undissolved crumb rubber particles and inorganic filler derived from dissolved rubber particles which could not be dispersed and stabilized by additive used in the bitumen-rubber system. At the same time the polyisoprene content of these crumb rubbers were low therefore only a limited content of polyisoprene could be dissolved by bitumen. This is the reason why only a weak elastic network could originate although it would be suitable for stable bitumen-rubber system.
Beside the poor storage stability it was found that these samples had relatively small softening point values compared to original bitumen 50/70 (actual crumb rubber samples: CR-02, CR-03, CR-05, CR-06, CR-07, CR-08). The reason is the relatively high content of extender oil causing softening effect in the bitumen-rubber system. Viscosity values also proved the softening effect compared to CSRB samples containing crumb rubbers having compatibility factor above 1.5. Summarized it was determined that higher polyisoprene content (least 25 wt%) and compatibility factor (least 1.5 wt%) can result better quality of CSRB.
Correlation was not observed between carbon black, total rubber hydrocarbons, chloroform extract of crumb rubbers and investigated properties of CSRB. This study does not deal with aging resistance of CSRBs although it has important positive effect on it because of carbon black derived from dissolved rubber particles.

4. PLANT PRODUCTION OF CSRB AND USING FOR ASPHALT MIXING AND ROAD CONSTRUCTION

The chemically stabilized rubber bitumen (CSRB) can be produced in the existing bitumen modifier units in the crude oil refinery using the newly developed improved wet process together with the multifunctional additive [25]. (Generally the conventional production method of rubber bitumen needs special equipments).
In these plants during the blending process intensive dissolution of the crumb rubber can be achieved in the bitumen, and by applying multifunctional additive the phase separation of the disperse system can be minimized. The high viscosity of the classic rubber bitumens [27], which causes issues quite often, can be reduced to the level of existing polymer modified binders by applying suitable technological parameters.
Emission problems and potential effects on health of originating gases have emerged in association with rubber bitumen production and paving application [20, 21, 28]. Certain measurements proved that concentration of gases emerging in asphalt mixing have not exceeded the one during normal asphalt mixing [21, 28]. Other studies, although, have reported higher emission rates compared to normal asphalt mixing [20]. It can be stated that the lowest temperature possible is recommended for minimizing the emission. However, in most cases it is complicated because of high binder viscosity.
Contrary to this fact the developed new process uses high temperature in the refinery, which temperature can reach 220°C. Gases emerging during crumb rubber and bitumen blending at high temperature are sucked and treated (repealed) by refinery. As a result this environmental issue can be solved by the refinery and does not rise at asphalt mixing and paving. The plant production process can be followed up by continuously viscosity measuring. When the viscosity is on the required level blending process should be stopped and product pumped to the product tank.

In Hungary and Slovakia several tests wearing courses were constructed using asphalt mixture containing CSRB. Crumb rubbers applied for plant production were selected by rubber analytical results presented in Chapter 3. Crumb rubbers used for production have compatibility factor between 2.0-2.5 signed by CR-12, CR-17 and CR-23 in Table 1. It is important to emphasize that every batch contained 25 tons had very similar performance and properties compared to samples prepared in laboratory. Figure 3 a-d presents AC-11 wearing courses paving at four different places in Hungary.

Figure 3 a-d: Asphalt wearing courses paving with AC-11 mixture containing CSRB

By using the conventional equipments no engineering problems were observed during asphalt mixing, transportation, spreading, compaction and rolling. It was also highlighted by the pavers that despite the low ambient temperature (6-7^{o}C) of paving tests section shown in Figure 3c, the asphalt mixture was easy to handle.
Asphalt mechanical tests were also carried out using CSRB derived from plant production. The aim was to compare the performance of asphalt sample containing CSRB with asphalts containing 50/70 penetration grade bitumen and 25/55-65 polymer modified bitumen separately. Binder properties applied for asphalt tests are shown in Table2.

Table 2: Properties of binder used for asphalt tests

Property	**Test Method**	**CSRB**	**50/70**	**25/55-65**
Softening Point, ^{o}C	EN 1427	59.0	51.0	74.0
Penetration at 25^{o}C, 0.1mm	EN 1426	65	58	45
Fraass breaking point, ^{o}C	EN 12593	-24	-12	-14

Substantially better performance was observed in all measured parameters on asphalt samples produced with the use of CSRB compared to asphalts containing 50/70 neat bitumen (Table 3). It can be concluded that CSRB performed on the similar level than that of polymer modified bitumen (Table 3). Measured values of permanent deformation were on the same level. Its low temperature performance proved better although fatigue characteristic and water sensitivity was slightly weaker than asphalt containing polymer modified binder.

Table 3: Comparison of three AC-11 type of asphalts produced with different binders

	Requirement in Hungary	**CSRB**	**50/70**	**25/55-65**
Binder content, wt%		5.1	5.1	5.1
Water sensitivity (EN 12697-12)				
$\frac{Tensile\_sterngth\_of\_wet\_specimen}{Tensile\_sterngth\_of\_dry\_specimen} * 100\%$	min. 80	93	86	95
Permanent deformation /Wheel tracking (EN 12697-22)				
Relative deformation, %	max. 5.0	1.0	3.6	1.0
Asphalt cracking*				
Cracking temperature, ^{o}C	max. -18.0	-31.0	-18.0	-24.8
Resistance to fatigue (EN 12697-24)				
E_{hhmax}, (N=10^{6}), microstrain	to be reported	188	143	201

* Thermal Stress Restrained Specimen Test (TSRST) [28]

Following the successful plant production together with paving experiences and the favourable asphalt mechanical tests CSRB application would be an effective way for road construction and pavement maintenance. Production and application of CSRB significantly supports the sustainability because used tyres utilization will be realized avoiding the stockpiling and burning of tyres.

5. SUMMARY

A new rubber bitumen process has been elaborated by which Chemically Stabilized Rubber Bitumen (CSRB) can be produced in the crude oil refinery. As a result of this research a quality requirement of crumb rubber was developed to determine which type of waste rubbers and crumb rubber can be used for rubber bitumen production having constant quality and suitable characteristics, primarily storage stable, suitable viscosity and softening point. It was found that higher polyisoprene content (least 25 wt%) and compatibility factor (least 1.5 wt%) of crumb rubber having particle size between 0-1.5mm can result suitable quality of CSRB. According to asphalt mechanical test results it can be concluded that CSRB performed on the similar level than that of polymer modified bitumen. Hot mix asphalt was produced using of CSRB and wearing courses paved in Hungary and Slovakia without any difficulties. In the near future further road section will be constructed and other asphalt mixture type will be tested on field to get more information about CSRB performance.
These experiences proved that CSRB means a valuable binder for pavement construction. A binder of such quality may promote general use of rubber bitumens for road construction and maintenance and may contribute to the improvement of the road systems.

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