Thermomechanical behaviour of bituminous materials including RAP and rejuvenator and Environmental impact of their fabrication process

Teză de doctorat – Rezumat

pentru obținerea titlului științific de doctor la
Universitatea Politehnica Timișoara
în domeniul de doctorat Inginerie civilă și instalații

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luna Iunie anul 2021

The public road network in Romania provides motorized access in most regions, localities of the country and according to the National Institute of Statistics [1] the length of the public road network at the end of 2019 measured 86 391 km from which: 17 873 km (20.69%) highways and national roads, 35 083 km (40.61%) county roads and 33 435 km (38.70%) local roads. The national roads represent the majority network of the country, approximative 70% of road traffic takes place on them. Based on the rehabilitation and modernization program that was performed over the past years in Romania, at the end of 2019 the national roads reached 95.1% roads with modernized surface course, 4% roads with semi-permanent surface course (macadam), 0.8% cobblestone roads and 0.1% dirt roads. Only 42.3% of the county roads and 19% of the local roads presents a modernized surface course. On the other side, only 62% of the total public roads did not exceed their service life. Therefore, by analysing all the presented data it can be concluded that a national program based on sustainable policies of rehabilitation and modernization of the Romanian's public road network is welcome in the near future.

It is important to mention that in Romania there are no policies regarding the reuse and/or recycle of materials in road domain even if there are some guidelines and some norms regarding the recycling methods. A national strategy regarding the reuse of the materials obtained by milling the old existing road pavements that exceeded their service life in the idea of producing higher-performance bituminous mixtures, is still missing. A proper strategy could be to impose the use of a minimum percentage of Reclaimed Asphalt Pavement (RAP) in the production of new bituminous mixtures. Such a strategy will lead in time to other strategies related to the increase of the amounts of RAP materials in the idea of producing higher-performance bituminous mixtures ('up-cycling'). This up-cycling solution could be a key factor in the sustainable development of Romania's public road network.

A proper strategy of re-using the RAP materials, based on the experience of other European countries and the European recommendations will become a key tool for Romania. The sustainability principles such as increasing the re-used and recycled content in products by ensuring their performance and safety, enabling remanufacturing, high-quality recycling, reducing environmental footprints, restricting single-use and improving product durability, reusability, upgradability and reparability should be accomplished (European Circular Economy Action Plan, 2020).

Over the last decades, many research efforts were spent to find better solutions to increase the amounts of recovered/recycled and renewable materials used in the road construction industry.

Roads, as all the other types of constructions require during their lifetime or at the end of it, several interventions such as rehabilitation, modernization, resurfacing, reconstruction, etc. in order to ensure the safety and comfort of the traffic participants. The Reclaimed Asphalt Pavement (RAP) refers to the term used for the material obtained by milling or by full-depth removal of the old asphalt pavement. The RAP material is considered 100% recyclable and its use in the production of new asphalt mixtures leads to important benefits as cost reduction or conservation of energy, virgin aggregates and binders, etc. [2], [3].

Most of construction materials can be only recyclable. On the other side, the asphalt is one of the few construction materials that is 100% reusable. Therefore, asphalt materials are considered as 100% reusable and recyclable materials. According to the Technical Briefing (2020) of the European Asphalt Pavement Association (EAPA) 'the reuse of the existing road material shall always be the first option and the recycling the second one' [4].

For economic and environmental reasons, the use of Reclaimed Asphalt Pavement (RAP) in the production of new bituminous mixtures HMA (Hot-Mix Asphalt) [2], [5]–[8] and WMA (warm-mix asphalt) [9], [10] has become a common strategy for the construction and maintenance of roads in many countries. During recent years, many countries have developed various policies based on the sustainability in the road domain, based on the recovery, the reuse, the recycling of materials in order to produce new eco-friendly materials.

According to the Annual Technical Report from 2018 in USA the asphalt industry is considered 'the most diligent recycler', where more than 99% of RAP material is being reused and 82.2 million tons of RAP material are used in new bituminous mixtures [11].

On the European side, according to the European Asphalt Pavement Association (EAPA) the amount of RAP material available in various European countries, at the end of 2018, was 49.50 Mt. The highest percentages of RAP material correspond to Germany (26% of total available RAP in 2018 in Europe), Italy (18%), France (16%), Great Britain (12%) and 28% for the other countries.

According to these data, in 2018, in Europe, near to 76% of the total available RAP material was reused in the production of a new hot/warm/cold bituminous mixture (71% hot or warm mix asphalt + 5% cold mix asphalt), 20% recycled of total and 4% used in other applications or put to landfill. However, the reuse of RAP in the production of new bituminous mixtures is not very popular in Romania as there is no reporting data regarding the reuse of such materials.

Various studies highlighted that when more than 20% of RAP material is used in new asphalt mixtures it lead to an increase of complex modulus and to deteriorations on fatigue life of final mixtures [7], [8], [10], [12]–[14].

In order to increase the amount of RAP material and to improve the mix characteristics, many studies showed the potential of rejuvenating agents to regenerate the hard-aged RAP binder and finally to induce a positive effect on the mechanical characteristics of the final product which can be considered as an eco-friendlier bituminous mixture [15]–[17].

The use of rejuvenators (Rej) was first introduced in 1960 as preservation treatment of pavement with the primary role to restore the physical and chemical characteristics of the hard-aged RAP binder.

The conclusions of several studies and the purposes of several ongoing research programs that are focusing on the investigation of the influence and the effect of various RAP materials and different rejuvenators on the final performances of the mixtures can be summarized as follows:

- rejuvenators can improve the permanent deformation of final bituminous mixtures - conclusions of Haghshenas et al. (2016) [18], Zhou et al. (2015) [19];
- the long-term performance of rejuvenators may not exhibit stability when exposing to

high temperature for a long period of time – conclusion NRRA report (2020) [20];

- rejuvenators can reduce the moisture resistance - conclusion of Haghshenas et al. (2016) [18], Tran et al. (2012) [21], Hajj et al. (2013) [22], Im and Zhou (2014) [23];
- rejuvenators can reduce the stiffness of the final mixtures – conclusions from Tran et al. (2012) [21], Hajj et al. (2013) [22], Im et al. (2014) [23];
- rejuvenators may improve the resistance to cracking of the final mixtures – conclusions of Tran et al. (2012) [21], Hajj et al. (2013) [22], Im et al. (2014) [23];
- rejuvenators improved the fatigue resistance and they can provide fatigue resistance even after expended hours of aging – conclusions from NRRA report form 2020 [20];
- low temperature cracking susceptibility of mixtures produced with RAP material is improved when rejuvenators were used. Also, even if the mixtures were subjected hours of aging, this process did not cause an important effect on the low cracking temperature – conclusions from NRRA report form 2020 [20].

The study presented in this thesis has been carried out within a collaboration between Politehnica University Timisoara/Faculty of Civil Engineering/Department of Overland Communication Ways, Foundations and Cadastral Survey from Romania and Université de Lyon/École Nationale des Travaux Publics de l'État (ENTPE), laboratoire de Tribologie et Dynamique des Systèmes (LTDS) from France. The objectives are, i) the characterization of the thermomechanical performances of binder blends and bituminous mixtures produced with Reclaimed Asphalt Pavement (RAP) and rejuvenator (Rej) and, ii) the investigation of the potential environmental impact related to the production of a mixture containing different amounts of RAP material and Rejuvenator.

Therefore, comprehensive experimental investigations were performed on binders and mixtures. All tests on binders and mixtures were performed in the Road Laboratory from Politehnica University Timisoara, together with the environmental impact assessment. On the other hand, the analyses, estimations and predictions of most parameters/characteristics of binders and mixtures were performed at ENTPE.

The study on binders (Chapter 2) focused on the properties of different binder blends produced by mixing one type of fresh binder (a straight run 50/70 pen. grade), a RAP-extracted binder and a rejuvenator of vegetal origin. The effects of RAP binder and rejuvenator were first analysed in terms of European conventional properties (penetration at 25°C, ring and ball temperature, Fraass temperature, density and elongation at 25°C). Their effects were also analysed on the thermo-rheological properties obtained from complex shear modulus tests (at intermediate and high temperatures) and BBR tests (at low temperatures). Linear viscoelastic (LVE) behaviour obtained from complex modulus tests were successfully modelled using the 2S2P1D (2 Springs, 2 Parabolic elements and 1 Dashpot) model developed at ENTPE.

The experimental plan includes a total of 17 binders consisting of the 50/70 fresh bitumen with and without the RAP-extracted binder with and without rejuvenator. Blending proportions between RAP binder, fresh bitumen and rejuvenator were calculated in order to reproduce ratios between these components within corresponding tested bituminous mixtures (with a 5.6% total binder content, the rejuvenator was not considered as part of it) containing 25%, 50%, and 75% RAP material and 0.00%, 0.20%, 0.40% and 0.60% of rejuvenator by mass of RAP material (a total of 12 blends). These proportions for mixtures gave 0%, 5%, 10% and 15% of rejuvenator by mass of the RAP binder in the blends. Three other blends between RAP binder and rejuvenator were considered (RAP + 5% Rej, RAP + 10% Rej, RAP + 15% Rej). These additional blends were produced and tested considering that they were used as 'base materials' in the estimation approaches presented in following sections. Also, the two base binders were tested.

The experimental results show a decrease in penetration and an increase in softening point, Fraass breaking point temperature and penetration index for the increase of RAP binder content and decreasing rejuvenator content in blends. Similar tendencies to those observed for the Fraass breaking point temperature and the penetration index values were found for the values of density at 25°C. For the binder blends produced without rejuvenator the elongation is decreasing with the increase of the RAP binder content and is always lower than 150 mm.

DSR complex modulus tests were performed from 25°C to 85°C and frequencies from 0.1 Hz to 10 Hz. The Time-Temperature Superposition Principle was validated for all tested binders. Data obtained with the DSR tests for all binders were successfully modelled by using the 2S2P1D model for which four parameters were considered constants for all tested binders and only three parameters were investigated. The analysed parameters and shift factors show some remarkable tendencies (linear or logarithmic trend) with the increase of RAP binder and rejuvenator contents in the final blends.

Steady shear viscosity at 85°C was obtained as the norm of complex viscosity at high temperature/low frequency in the domain of Newtonian behaviour of binders. Steady shear viscosity values at temperatures from 75°C to 25°C were calculated from the experimental values obtained at the reference temperature of 85°C. For all temperatures, steady shear viscosity values are increasing with the increase of RAP binder content and with the decrease of rejuvenator content in the blends. The addition of the rejuvenator was observed to counterbalance the stiffening effect of the RAP binder in the blends.

High and low critical temperatures were determined from the data obtained with the DSR and BBR tests. The tests are in agreement with the overall Superpave framework but some minor differences in the analysis of test results were applied to obtain the considered critical temperatures of the tested binders. The experimental results show that both critical temperatures increase with the increase of RAP binder content in the blends, and they decrease with the increase of Rej content within blends. It seems that the rejuvenator can have a counterbalancing effect as it neutralizes the effect of the RAP binder. However, it is interesting to notice that a lower dosage of rejuvenator is necessary to obtain a similar low critical temperature as the one of the 50/70 binder than to meet the high temperature specifications.

For most of the analysed parameters, the results obtained for the blends produced with 10% or 15% rejuvenator by mass of the RAP binder, are closer to those obtained for fresh binder 50/70, independently of the RAP binder content. The 10% or 15% rejuvenator dosage depends on the investigated parameter. Practically, this indicates the capability of the rejuvenator, in terms of mechanical characteristics, to rejuvenate the hard-aged RAP binder and finally to provide a final product with similar properties of fresh binder.

Strong relations were observed between the experimental results obtained for the conventional parameters, steady shear viscosity at 25°C and both critical temperatures.

Two different estimation methods were proposed, and all investigated parameters were estimated for all binder blends from experimental results obtained for the base constituents.

The first estimation method is a classical approach in which 5 binders are considered as base materials (fresh binder, RAP binder and the three RAP + Rej blends).

The second estimation is a new three component approach (fresh binder, RAP binder and rejuvenator). For the rejuvenator some equivalent values for all parameters were obtained by minimizing the distance between experimental and estimated values for all binder blends. It should be highlighted that these equivalent values were used only in the context of the blending law, therefore they are not intended to reflect actual properties of the rejuvenator.

Slightly better correspondence was found between the estimated values of all above-mentioned parameters obtained with the 2nd estimation, which is an original input of this work, and experimental values. Moreover, the 2nd estimation has the great advantage to only require parameters for each of the three base constituents when the dosage if fixed.

In order to obtain phase angle from the estimated values of the norm of complex modulus for all binder blends over the whole frequency and temperature domain, the 2S2P1D model was calibrated on master curves of the norm of complex shear modulus at a reference temperature of 55°C in order to be sure that all analysed binders are thermorheologically simple. This is a new method which was proposed in this research. The determined values of the phase angle are close to the measured values for all binder blends. A better correlation with the experimental measurements was found for the phase angle values calculated from the 2S2P1D fitted on the master curves of the norm of complex shear values obtained with the 2nd approach.

The statistical analysis performed in order to highlight the accuracy of both estimation methods for all parameters, considering all binder blends, shows that a more accurate estimation resulted by applying the second estimation method.

A new method was proposed if the equivalent parameters for the rejuvenator are not known from previous experiments, a good approximation can be obtained from only one test performed on the binder blend produced with the RAP binder and the maximum rejuvenator content: RAP + 15% Rej in the case of this study. In these conditions, the results used for the approximated values for the rejuvenator are very similar in comparison to the ones considering the optimized values. This conclusion is also validated on two new binder blends having other rejuvenator contents of 7.5% and respectively 8.5%. This proposed approach presents the great advantage of requiring less testing than the classical approach and can be used for any combination of RAP/rejuvenator, in contrast to the classical approach.

The equivalent parameters for the rejuvenator are probably only rejuvenator dependent, which is another advantage of the proposed method. This point should be confirmed with a wider range of fresh and RAP binder types.

The study on mixtures (Chapter 3) focused on the investigation on 13 bituminous mixtures produced with different amounts of RAP material with or without a vegetal origin rejuvenator. The considered fresh and RAP binders and rejuvenator, as well as their proportions, are the same than the ones used for the binder blends experimental campaign. All the materials used in this study are specific for Romania.

All materials (aggregates, binders and RAP material) were characterized with a series of tests according to the specifications from the Romanian Standards. First, seven types of HMA were produced and tested in order to determine the optimal binder content. Then, a second campaign tested 13 types of mixtures produced with RAP material and rejuvenator that have a similar continuous 16 mm grading curve as the reference/conventional HMA, the same total binder content (5.60% total binder content by weight of the final mix not including here the rejuvenator) and the same RAP material (25% RAP 0-8 + 75% RAP 8-22.4).

Hydrostatic measurements, Marshall tests, indirect tension tests at different temperatures (10°C, 15°C, 20°C and 25°C), cyclic compression tests with confinement (300 kPa, confinement 50 kPa, 50°C, 10000 cycles) and two-point bending complex modulus tests on trapezoidal samples were performed. Influence of RAP material and rejuvenator on the behaviour of mixtures was highlighted. The effect of increasing the RAP material content conduced to a stiffer behaviour of the bituminous mixtures. On the other hand, the increase the rejuvenator content conduced to a reverse effect, which is counterbalancing the effect of the RAP material.

It is important to notice that the binder blends of the different mixture types are the same than the ones tested in Chapter 2. Then the general effect of binder on mixture properties could be a focus of the study. However, it is important to mention that the binder blends investigated in Chapter 2 were perfectly blended in the laboratory. Even if the percentages between fresh, RAP binder and rejuvenator are the same, some differences between these perfect blends (Chapter 2) and the blends from the mixtures can occur.

For most of the investigated parameters (Marshall characteristics, stiffness modulus and dynamic creep), their variations with the penetration of the corresponding binder blends resulted in strong relationships for the mixtures/binder blends produced with the same RAP material/binder content (25%, 50% and 75%). However, the overall relation between these parameters and the penetration is not so strong. These tendencies could be explained by the fact that most probably the use of the RAP material leads to a change in the grading curves of the final mixtures. Besides, the use of the same compaction energy for all mixtures, the rejuvenator content which was established as a function of the mass of the RAP material and the difference between the density of the RAP aggregates and the density of the virgin aggregates are all leading to scattered results.

Experimental data obtained from the complex modulus tests were fitted with 2S2P1D model. It was observed that 2S2P1D parameters and the shift factors of mixtures do not appear to follow a clear tendency with the increase of the RAP material and rejuvenator contents. This observation is in contrast with the linear tendencies of these parameters with the RAP binder content which were observed for the corresponding binder blends. The glassy modulus of mixtures presents a satisfactory relation with the penetration of the corresponding binders.

SHStS (Shift, Homothety, Shift and time Shift) transformation proposed by ENTPE team was applied to predict mixtures behaviour from corresponding binder blend. A satisfactory prediction was obtained despite the limited range of the DSR measurements on binders. 2S2P1D model was also successfully used to predict LVE behaviour. A linear relationship was obtained between some parameters of the mixtures, including the norm of complex modulus at 15°C and 10 Hz, and the penetration values of the corresponding binder blends.

The norm of complex modulus at 15ºC and 10 Hz of the bituminous mixtures were simulated from their corresponding binder blends behaviour and with the three SHStS constants. A good correlation between the experimental and simulated results of the norm of complex modulus at 15°C and 10Hz was obtained, proven by the satisfactory R^2 value of 0.981 and by the relative errors which are always lower than 5%.

It worth mentioning that all tendencies presented in Chapter 3 partially confirms the conclusions drawn within Chapter 2.

An Environmental Impact Assessment (EIA) was performed (Chapter 4) in order to estimate the potential environmental impact related to the production process of 13 types of bituminous mixtures, defined by the following three stages: raw material supply, transport and manufacturing, of one tonne (1 T) of the considered bituminous mixtures while all the other processes were assumed to be similar. EIA was performed by using GaBi software. A comparative and an internal analysis were performed in order to better highlight the environmental impact of the production process of bituminous mixtures containing RAP material and rejuvenator.

As expected, the production process of the conventional HMA mixture leads to the highest environmental impact. The addition of RAP material leads to a clear decrease of the energy use and environmental impact. On the other side, when rejuvenators are used in this process, they can affect the energy balance and reduce the difference in environmental impact. However, the increase of the rejuvenator content corresponds to a small increase of the impact. A considerable impact was observed for the land use, due to the fact that the rejuvenator considered in this study is a mix of vegetal oils.

When less than 25% of the considered RAP is used in the production of a new bituminous mixture, the characteristics of the final mix could be considered within the limits specified in the Romanian Standards for a bituminous mixture with a 16 mm maximum aggregate size. Therefore, the decrease of the environmental load is close to 20% for this solution compared to the conventional one.

When high amounts of RAP are used in the production of a new bituminous mixture, rejuvenators should be used, too. Although the solution with 75% RAP (D.75.R.0) was considered to highlight the environmental impact potential of this 100% recyclable RAP material. A substantial decrease of the environmental impact as more than half of the conventional solution could be obtained.

For the production process of bituminous mixtures containing RAP material and different amounts of rejuvenator, an increase of the environmental impact was observed with the increase of the rejuvenator content compared to those produced with RAP material only. However, this only represents approximately 1.5% of the environmental load increase per 0.2% rejuvenator content by the total mass of the final bituminous mixture.

Based on the experimental work and on the results presented in Chapters 2 and 3, the mix design D.50.R.0.4/the corresponding binder blend (50/70 + 50% RAP + 10% Rej) presents a closer behaviour to the one of the conventional bituminous mixture/the corresponding fresh binder (50/70). The environmental impact for this solution (D.50.R.0.4) leads to a decrease of approximately 33-40% (depending on the analysed indicator) than the conventional HMA.

Several points of the study are worth further investigation. Therefore, as perspectives the following points could continue the work developed in the presented thesis:

- the proposed method (2^{nd} approach) to estimate the conventional properties, LVE properties, steady shear viscosity, norm of complex shear modulus and critical temperatures of binder blends produced with RAP binder and rejuvenator, is promising but still needs validation on a larger number of different binders and rejuvenators;
- a future investigation should be carried out by performing DSR tests on a larger temperature and frequency ranges and also by performing an investigation at low temperature of the aged (RTFOT or PAV aging) binder blends. Also, a study of the linearity could be performed;
- another important aspect that should be investigated is related to the relation between the composition in terms of chemical properties and microstructure and the 2S2P1D parameters of binder blends that are produced with RAP binder and rejuvenators. This could bring information on the chemical and thermomechanical effects of the rejuvenator on the long-term behaviour of binder blends;
- different blending conditions should be taken into consideration in a future work in order to compare the estimated results with the experimental data;
- regarding the study of the thermomechanical behaviour of mixtures produced with RAP material and rejuvenator, further research could be carried out on fatigue, rutting and thermal cracking in order to investigate the effect of the rejuvenator on these characteristics. Also, complex modulus tests could be investigated over a larger temperature and frequency ranges by performing tension-compression tests;
- further analysis should be carried out on the relations of mechanical properties between binders and mixtures. In particular, correspondence between fatigue performances and rutting performances of mixtures and the corresponding binder properties could be investigated;
- an important point that worth investigation is the comparison between the behaviour and properties of corresponding binder blends, mastics, and mixtures. This research could lead to some interesting data regarding the self-healing of cracks and the recovery of the linear visco-elastic properties;
- continuing the analyses on environmental impact for the solutions presented in this study, a Life Cycle Assessment could be made considering all the other processes excluded by the system boundaries in this study. Thus, the Life Cycle Costing (LCC) analysis could complete the LCA for mixtures produced with RAP material and rejuvenator by considering the local costs and materials;
- all conclusions that were drawn in this study are based on laboratory measurements and related analyses. Therefore, a validation with in-situ through observations regarding the performances of mixtures should be performed.

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