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Road and Rail Infrastructure V

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ANALYSIS OF BITUMEN AGEING PROCESSES AND INNOVATIVE TECHNIQUES FOR THE REJUVENATION

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Abstract

Bitumen (asphalt) oxidative aging is one of the prevalent causes of the pavement distresses which results asphalt pavement susceptibility to fatigue and low temperature cracking. Ultimately, aging of asphalt leads to pavement failure after years of usage. Environmental and financial issues raise demand of effective pavement materials recycling. However, aged asphalt materials re-usage is complex and specific. The idea of the research is to insert nanomaterials to aromatic rejuvenators for aged bitumen mechanical and physical properties regeneration. There are different types and chemical composition nanomaterials as products for new bitumen modification, this research focus on aged bitumen regeneration and prolonging the whole service-life of asphalt. This paper presents comprehensive literature analysis and explicit design of experiment subjected to bitumen (asphalt) ageing, rejuvenation and bitumen regeneration using nanomaterials. This innovative research allowed investigating the phenomenon of asphalt rejuvenation using nanomaterials and identifying the most appropriate rejuvenating additives in asphalt binder.

Keywords: bitumen oxidation, bitumen ageing, rejuvenation, nanomaterials

1 Introduction

1.1 Bitumen ageing process

The aging of bitumen is one of main factors influencing the lifetime of an asphalt pavement. The process of aging involves chemical and physical property changes that usually make bituminous materials harder and more brittle, thus increasing risk of pavement failure which results asphalt pavement susceptibility to fatigue and low temperature cracking [1, 2].
In general, bitumen aging takes place in two stages, namely short-term aging at high temperature during asphalt mixing, storage and laying, and long-term aging at ambient temperature during in-service. The mechanisms of aging include oxidation, evaporation, polymerization and physical hardening [3]. Oxidative aging is an irreversible chemical reaction between components of bitumen and atmosphere oxygen. It may occur through different reactions, such as photo-oxidation and thermal oxidation, which changes the composition of the asphalt, creating a more brittle structure [1]. For bituminous binders, loss of volatile components (evaporation) is also considered as an aging mechanism [4]. Polymerization subjected to resins transform into asphaltenes, causing an increase in the brittleness of the asphalt along with a tendency toward non-Newtonian behaviour [5-8]. Blokker and Van Hoorn were the first claimed that the process which involves wax crystallization and asphaltene aggregation can be called as physical hardening phenomenon [9]. Physical hardening is a reversible process, which changes the rheological properties of bitumen without altering its chemical composition. At ambient temperatures, physical hardening normally is very slow, but it can speed up

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at low temperatures [10]. A lot of studies and investigations were done by S. Hesp analysing and understanding physical hardening phenomenon in order to predict cracking performance [1-3, 11, 12]. Using X-ray diffraction, he were able to show that binders with high wax contents invariably suffer from physical hardening [2].
Chemical changes during aging process have been studied widely in the past. It is known that oxidation of bitumen produces carbonyls and sulfoxides and increases polarity, causing increases in bitumen viscosity and softening point, etc. Chemical changes may differ largely between different bitumen, especially when they are from different crude oil and production technology [1, 6, 8]. Finally, aging of bitumen leads to pavement failure after years of usage. Therefore, environmental and financial issues raise demand of effective pavement materials recycling and use of rejuvenators in order to improve asphalt mixture resistance to ageing.

1.2 Bitumen rejuvenation methods

Bitumen rejuvenation process is a relevant topic related with reclaimed asphalt pavement (RAP) and hot-in place recycling (HPR) technologies, when aged bitumen properties can be more or less restored by adding a special amount and chemical composition rejuvenator. Lots of studies have been done on rejuvenating methods such as use rejuvenating agents in mixing them with aged asphalt mixes at high temperature or spraying it into the surface of asphalt pavement; Rejuvenator Seal Materials (RMS), which is used after 3-4 years of pavement construction [13] or modifying aged bitumen with aromatic oil, paraffinic oil, soft bitumen, and nano materials [14, 15]. During the rejuvenator process is very important to determine the appropriate type and amount of rejuvenator so that the low temperature properties of the mixture can be provided improved [16]. Summarized literature review of rejuvenator amount used to restore aged bitumen properties is shown in Table 1. Moghaddam and Baaj [17] analysed different types of rejuvenating agents in production of recycled hot mix asphalt. In his opinion, good rejuvenators should properly react with aged binder and produce high quality recycled binder. It is important to note, that over dosage of rejuvenator can cause other problems such as loss of adhesion and stripping of the asphalt film from aggregate. Simonen et al. (2013) explore bioflux solvent influence to bitumen ageing, chemical and rheological characteristics. The amount of solvent depend on regenerated bitumen SARA fraction content [4]. Researcher have found that rheological properties of cured and long-term aged bitumen using bioflux solvent was similar to fresh bitumen. Recently, the scientific interest has risen for exploring alternative binders and rejuvenators made from the recycled engine oil bottom (REOB), especially in Canada and North America. REBO contain chemical compositions similar to conventional asphalt binders, however the composition is inconsistent [12]. The use of REOB for asphalt mixtures is controversial, uncertain, and may accelerate physical and chemical hardening, leaving the binder brittle enough to cause cracking in the aspahlt pavement [11]. There are many other common alternative binders, such as bio-binder, palm oil, engine oil residue and etc., which are being investigated and analysed by other scientists [24, 25].

Table 1 Rejuvenator amount for restore aged bitumen properties

Rejuvenator	Content, %	Bitumen type	Researcher
650/900	21-33	Recovered	[18]
Aromatic oil + prepolymer shells	5	40/50	[19]
Organic blend	11	PG 64-22	[20]
Paraffinic base oil	18	PG 64-22	[20]
Aromatic extract	18	PG 64-22	[20]
Refined tallow	10	PG 64-22	[20]

Blomberg et al. (2016) investigated stability of bitumen 160/220 modified with nanoclay (organically modified montmorillonite, C20A) and polymeric diphenylmethane diisocyanate (MID). Researchers determine promising results adding 2 % of MID and 10 % C20A [21]. Modification of the origin (neat) binder with nano-SiO2 demonstrated significant improvements in physical and thermal properties with higher thermal stability and higher roughness. Selection of the optimum modifier content was based on Dynamic Shear Rheometer (DSR) asphalt fatigue and rutting tests as well as work of adhesion analysis [22]. Using nano ZnO, the fatigue life of the asphalt mixtures increased significantly, the modified ACs had greater adhesion energy with aggregate and showed greater fatigue life [14]. Adding Nano-clay in bitumen increased the viscosity of asphalt binders and improved the rutting and fatigue resistance of asphalt mixtures [21]. Addition of nano-silica into the control asphalt binder did not greatly affect the low-temperature properties of asphalt binders and mixtures [15]. In addition, a new approach for rejuvenation of aged bitumen was investigated using microcapsules (MicroWCOs) containing waste cooking oil. Waste cooking oil was easily penetrated into aged bitumen as a rejuvenator and recover its virgin properties, but was recognised uncertainly for long term performance [23].

1.3 Objective of paper

The main objective of this study is to review of the bitumen aging phenomenon and to present the design of experiment subjected to bitumen rejuvenation and regeneration process using nano additives.

Table 2 Variety of nano addictive for bitumen modification

Nano addictive	Addictive content in bitumen, %	Bitumen type	Researcher
Nano-SiO_2	4	PG-76 (70/100)	[26]
Nano-TiO_2	5	60/70	[27]
NanoClay	10	160/220	[21]
NanoClay+MDI	10+2	160/220	[21]
NanoClay+SBS+CR	3+2+1	PG 64-22	[28]
Nano-SiO_2	1	60/70	[29]
Nano-ZnO+SBS	4+4	PG 64-16 (50/70)	[30]
Nano-TiO_2 + Nano-SiO_2	5+2	50/70	[31]
NaP1 (syn. Zeolite)	5	35/50	[32]

2 Materials and methods

2.1 Materials

Bitumen binders. Investigation of innovative bitumen rejuvenation techniques is set for laboratory aged and recovered from pavement bitumen. Laboratory ageing will be done for neat 70/100 bitumen and PMB 45/80-55 modified bitumen. The recovered binders from RAP also correspond neat and modified binders used in wearing layers of asphalt pavement. For different rejuvenating agents will be used in this study:

- Paraffinic oil – extracts (petroleum), heavy paraffinic distillate solvent.
- Vegetable oil – edible vegetable oils are foodstuffs which are composed primarily of glycerides of fatty acids being obtained only from vegetable sources. They may contain small amounts of other lipids such as phosphatides, of unsaponifiable constituents and of free fatty acids naturally present in the oil.
- Aromatic oil – a distillation bottoms rich in indene, methylindenes and
- B650/900 (or similar) – soft penetration graded bitumen

According literature review three types of nano additive was selected: ZnO, TiO_2, and SiO_2. Before blending with rejuvenators, the most stable nano partilecs insert method will be determine: dry or dissolved in kerosene.

2.2 Methods

2.2.1 Bitumen recovery from aged asphalt mixture

Bitumen binder recovery method based on Nösler et al. (2008) research results [33]. Naturally aged bitumen extraction should be performed according standard procedures EN 12697-3 and EN 12697-1. Firstly, asphalt mixture is heated to 150 °C in an oven for 2.5 h. The loose asphalt mixture hold in methylenechloride for 8-9 h. Then an amount of mix is brought into the centrifuge and left to cool to 80 °C before starting the extraction. Extractions are continued until aggregates are clear and no binder is remaining in the wash drum. The binder is finally also recovered in the same way with the rotary evaporator according to EN 12697-3.

2.2.2 Bitumen ageing in the laboratory conditions

The rolling thin film oven test (RTFOT, EN 12607-1) and the pressure ageing vessel (PAV, EN 14769) perform to simulate the short term and long term ageing of bitumen. RTFOT bottles with 50 g of bitumen will be placed in carousel and oven at 163 °C, where hot air is periodically injected inside at a rate of 4000 ± 200 ml/min for 75 min. PAV is usually adopted to simulate the oxidation process that takes place during the service life of the pavement. 50±0.5 g of bitumen after RTFOT is poured in a pan with a diameter of 140 mm. The thickness of the film is about 3.2 mm. PAV will be performed at 90 °C and 2.1±0.1 MPa air pressure for 22 h.

2.2.3 Mixing method

Duplex assembly mixer used to ensure steady dispersion of nano particles in the rejuvenator, as well as modified rejuvenator even blend with aged bitumen binder. The nano particles blend with rejuvenator at 20±5 °C, insuring 3000 rpm mixing speed for 25 min. The assemble of modified rejuvenator and aged bitumen (reheated at 130 °C for 30 min) performed at 130 °C and 3000 rpm speed for 30 min.

2.2.4 Characterisation of bitumen chemical properties

Fourier transform infrared (FT-IR) spectroscopy was used to monitor the changes in the chemical composition of bitumen after ageing and quantify the oxidation products [34, 35, 37]. FTIR spectroscopy tracked the changes in specific compounds upon ageing for unmodified bitumen. During the study, the evolution of carbonyl and sulphoxide compounds was analysed from FTIR spectra after ageing of unmodified bitumen. In addition to the carbonyl and sulphoxide indices, other indices such as aliphaticity, aromaticity were also monitored. Infrared (IR) spectroscopy is applied to determine the functional groups in the organic molecule. Infrared spectrum at the appropriate wave length gives as an information about visible functional groups in bitumen such as sulfoxide, carbonyl groups, aromaticity. The bitumen samples for FTIR analysis were prepared by using 1 g of bitumen. Each sample was placed on a diamond and loaded a certain load to allow the sample to completely cover the area of the diamond. FTIR spectra of all samples were measured in the 4000–600cm^{-1}, at a resolution of 4 cm^{-1}, accumulating 24 scans per spectra. Thin Layer Chromatography with flame-ionisation detector (TLC/FID), the IATROSCAN MK-6s was used to separate bitumen into SARA fraction: saturates, aromatics, resins, and asphaltenes. The samples were dissolved in toluene with concentration of 1 % (w/v). After Chromarods were cleaned and activated, 1 μL of sample solution was spotted on each Chromarod using a spotter. Saturates were eluted with n-heptane (100 %), aromatics with solution of toluene (80 %) and n-heptane (20 %) and resins with solution of dichloromethane (95 %) and methanol (5 %). Finally, the Chromarods were scanned in the TLC-FID analyser and the average value of the readings was taken as the main result.

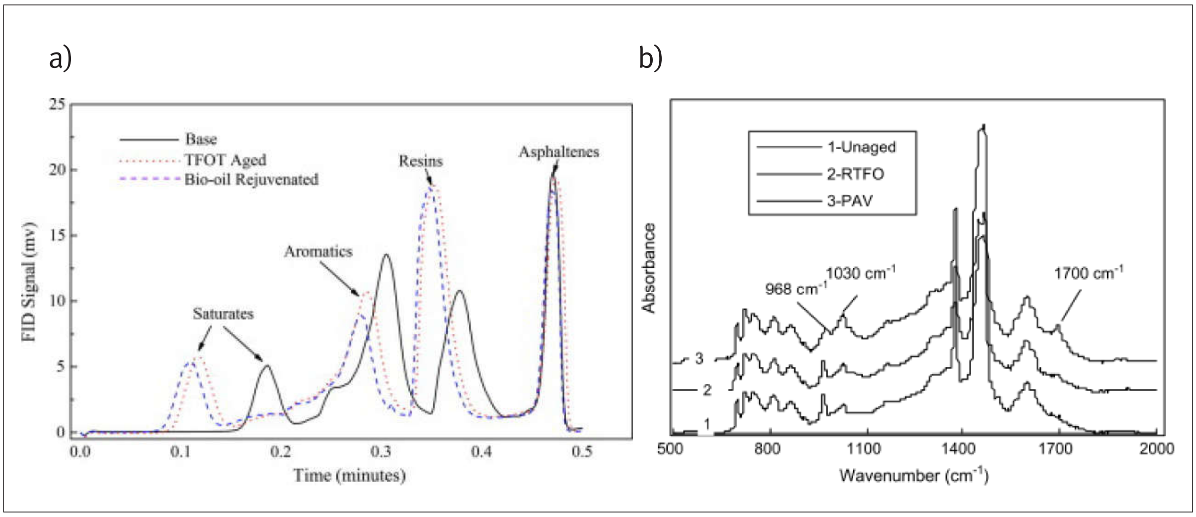

Figure 1 a) Example of SARA fraction spectra of virgin, aged and rejuvenated bitumen [36], b) Example of FTIR spectra of bitumen PG76 binders before and after aging [25]

2.2.5 Bitumen rheological properties characterisation

Dynamic Shear Rheometer (DSR) can be used for different rheological and mechanical properties evaluation. The optimal rejuvenation and nano addictive contents as well as their influence to bitumen will be determine over dynamic shear modulus G* and phase angle measured at 25 °C under strain of linear visco-elastic range at angular frequency of 0,1 rad/s. The complex modulus of rejuvenated binder will be determined performing frequency sweep test at temperature ranges from -34 to 80 °C, with 6 °C increments.
Bending Beam Rheometer (BBR) testing used for modified bitumen resistance to thermal cracking evaluation. Tests will be performed according to EN 14771.

3 Bitumen binder rejuvenation experimental plan and analysis methods

Due to complex research objective the experimental plan of bitumen binder regeneration and modification for long-life pavement project composed of three phases: Phase I. – determine the innovative bitumen rejuvenation method; Phase II – implement the innovative method for recovered bitumen regeneration; Phase III – simulate HPR techniques in laboratory to evaluate bitumen rejuvenation influence to asphalt mixtures.
The objective of phase I is to determine the effective/optimal rejuvenator and nano addictive types and their proportions for bitumen chemical and rheological properties regeneration. Experimental plan of innovative bitumen rejuvenation method determination is shown in Figure 2. For this research phase the 70/100 and PMB 45/80-55 bitumen were selected assuming that the majority of national and rural roads in Lithuania was paved with the common used mixtures.
The objectives of phase II is to proven an innovative bitumen rejuvenation method implementation for recover and naturally aged bitumen regeneration. Experimental plan of innovative bitumen rejuvenation method implementation is shown in Figure 3.

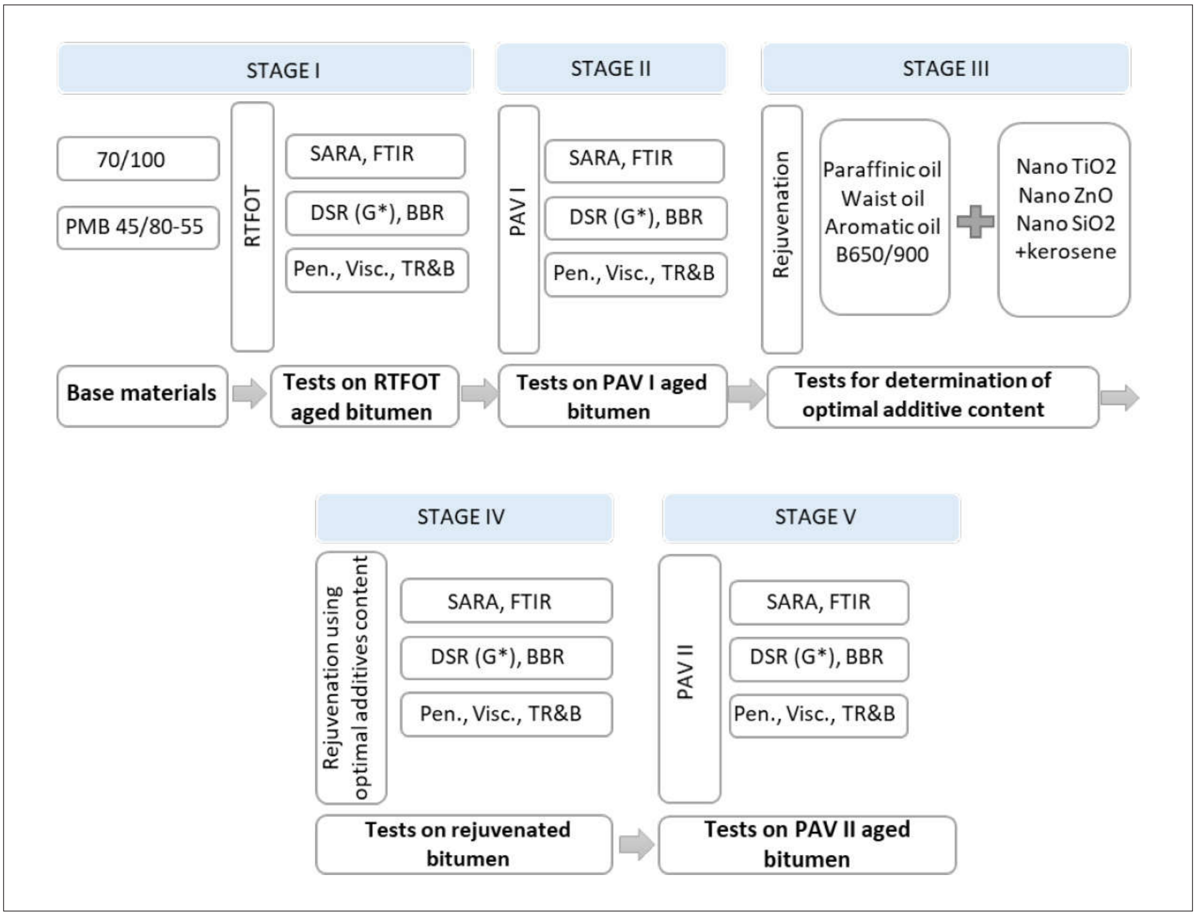

Figure 2 Experimental plan of innovative bitumen rejuvenation method determination

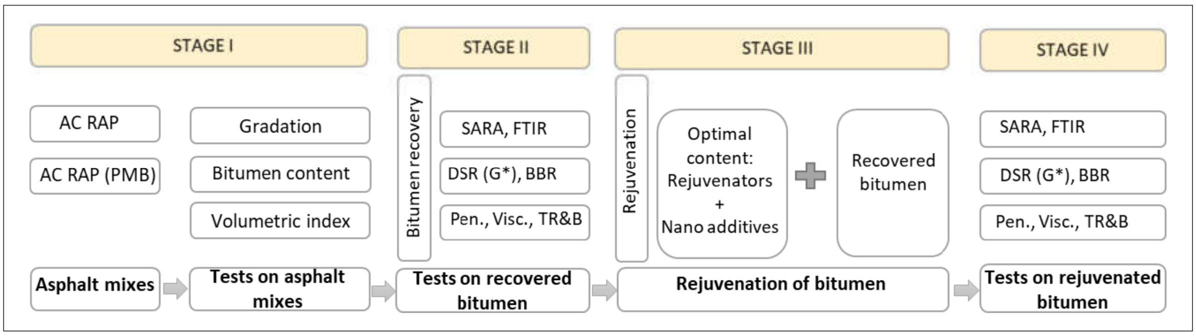

Figure 3 Experimental plan of innovative bitumen rejuvenation method implementation

Conclusions

Based on analysis the following conclusions can be drawn:

1) The mechanical properties of aged bitumen can be regenerated using rejuvenation agent, however the correct rejuvenator and its quantity have to be determined evaluating chemical composition.
2) The insert of nano-ZnO, nano-SiO_2, or nano-TiO_2 to bitumen can improve resistance to low-temperature cracking and permanent deformation, but the particles chemical composition influence, their size, blending methodology and stability have to justified.
3) Fulfilled three phases experimental plan will conclude scientifically based innovative bitumen rejuvenation method which will assure effective and durable hot-in place recycling of deteriorated asphalt pavement.

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