

## **INFLUENCE OF REJUVENATING ADDITIVES ON RECYCLED ASPHALT ( RAP ) PROPERTIES**

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### **ABSTRACT**

*Asphalt Industry continues to strive towards a more sustainable and environmentally friendly future. Their main focus is on reducing energy consumption and on reduced carbon foot-print. Currently there are a number of processes and products being discussed and tested.*

*All parties involved in the asphalt chain, are looking for possibilities to increase the percentage of RAP. However, they are not always paying attention to the impact on the binder properties and the final asphalt mix. In this project, the influence of different rejuvenating additives on the binder properties and the RAP performance will be investigated.*

*Among others, the addition of different virgin bituminous binders is compared to the addition of a specific hydrocarbon oil and several bio- oils.*

*The first phase of the project will measure by means of DSR the effect of rejuvenating additives on the binder, before and after both short and long term ageing. This paper will be reporting the results of this first phase. The next phase of the project will deal with the effect of the rejuvenated binders on the asphalt mix properties.*

**Keywords:** Rejuvenating, Oil, Recycling, DSR, PAV

## 1. INTRODUCTION

Asphalt Industry continues to strive towards a more sustainable and environmentally friendly future. Their main focus is on reducing energy consumption and on reduced carbon foot-print. Currently there are a number of processes and products being discussed and tested.

All parties involved in the asphalt chain, are looking for possibilities to increase the percentage of RAP. Currently 30-50% is being added into many asphalt mixtures and considered 'standard practice'. In some countries use of RAP goes up to 70 %. Sometimes even 100% reuse is claimed.

This is being driven by environmental and commercial factors. For environmental factors, one thinks about less CO<sub>2</sub> produced. RAP is only heated to about 110°C. Typical figures found in literature are 20 to 40 % less CO<sub>2</sub> during production. When maximising temperature reduction techniques, the benefit is even higher.

Concerning economics, less virgin bitumen is required, the higher the percentage RAP is used. Also the heating of the RAP versus raw materials is considered here.

All techniques currently used, are meant to "rejuvenate" the old binder in the RAP somehow and get the asphalt renewed.

For this, we find different types of rejuvenators used, claiming that the binder is completely renewed and the asphalt has regained its original properties.

However, little information can be found on the impact of the different additives on the binder properties and the final asphalt mix. And more over, the behaviour of the rejuvenated binder in the next 'ageing' steps.

Do the rejuvenated binders act or behave the same way as a 100% fresh binder after mixing, after compaction and during several years on the road?

In this part of the project, we have tested the influence of different rejuvenating additives available on the market, on the binder properties fresh, after RTFOT and after PAV, representing respectively the mixing step and the ageing on the road. As binder tests, base properties such as penetration and softening point are performed, this in addition to rheological tests (temp/frequency sweeps) with DSR.

A next step in the project will be the investigation of the RAP performance rejuvenated with the different rejuvenators in asphalt. Functional requirement tests such as stiffness, rutting and fatigue will be investigated. However, this part of the project is ongoing and is not reported in this paper.

## 2. INVESTIGATION DESCRIPTION - PROJECT OUTLINE

In this investigation, several types of rejuvenation known on the market or claimed to be fit for purpose are tested.

In the first place, the addition of softer virgin bituminous binder is investigated. This is probably the most used or known method. The old binder is upgraded through the addition of new virgin binder, with the goal to have in the new asphalt mix, the 'standard' used grade for that specific type of asphalt. E.g. a RAP binder with pen 20 needs to be blended with a softer pen grade, to target a final 35/50 grade in the mix for binder courses. Depending on the percentage RAP used in the mix, this can be a 50/70 grade or 70/100 grade. This is to be calculated by using the penetration blending rule.

As asphalt contractors do not always have different storage tanks available, to differentiate between and store different grades, "easier" solutions are searched.

A first example is the addition of specific hydrocarbon oil. The type used in this project is defined as liquid process oil, conform all current REACH and PAH limits. It has a typical viscosity at 40°C of 700 mm<sup>2</sup>/s.

Besides the hydrocarbon oil, we also tried the addition of several bio oils.

One type was a vegetable oil. It is 100 % originated from plants and not harmful. The product is not classified as dangerous according to Directive 67/548/EEC and its amendments and according to EU legislation.

The second type is a tall oil distillate. Tall oil originates from Pine trees. The oil is described to be used as an emulsifier for oil extended SBR latex and for metal working oils, as a base for alkyd resins, as an anionic floatation reagent, and as an asphalt emulsion additive. The product is not classified as dangerous according to Directive 67/548/EEC and its amendments and according to EU legislation. Classification and labelling have been performed according to EU Directives 67/548/EEC and 1999/45/EC (including amendments) and the intended use.

Further in the paper, in tables and graphs the different additives, binders and tests will be indicated as:

- RAP : recovered binder from the reclaimed asphalt pavement
- PG35/50 : pure, virgin binder – pen grade 35/50
- VB : virgin binder ; pure (soft) binder pen grade 160/220
- VO : vegetable oil
- HO : hydrocarbon oil
- TO : tall oil
- Fresh : non- aged binder or blend
- RTFOT : rotating thin film oven test – short term aged binder
- PAV : pressure ageing vessel – long term aged binder.

All rejuvenator types are added to the recovered RAP binder in such amount to target a penetration grade 35/50 (specific pen target 43).

The RAP material is a typical Dutch dense asphalt concrete mix, with a 0/16mm grading and initially 70/100 pen grade bitumen from over 15 years old.

### Test description

First step was to make an experimental blendline with fresh binder, similar to the pen of the recovered binder (25dmm). This to get an idea of how much rejuvenating additives to add to get to a refreshed binder. The target penetration of this refreshed binder was chosen to be a 35/50 penetration grade bitumen with a pen of 43 dmm.

The binder for these blendlines had 27 dmm penetration.

**Table 1: Results of experimental blendlines**

	pen 27	1% TO	2% TO	1% VO	2% HO	4% HO
pen dmm	27	36	46	43	36	42
R&B °C	56.8	55.0	53.6	52.6	54.5	54.2

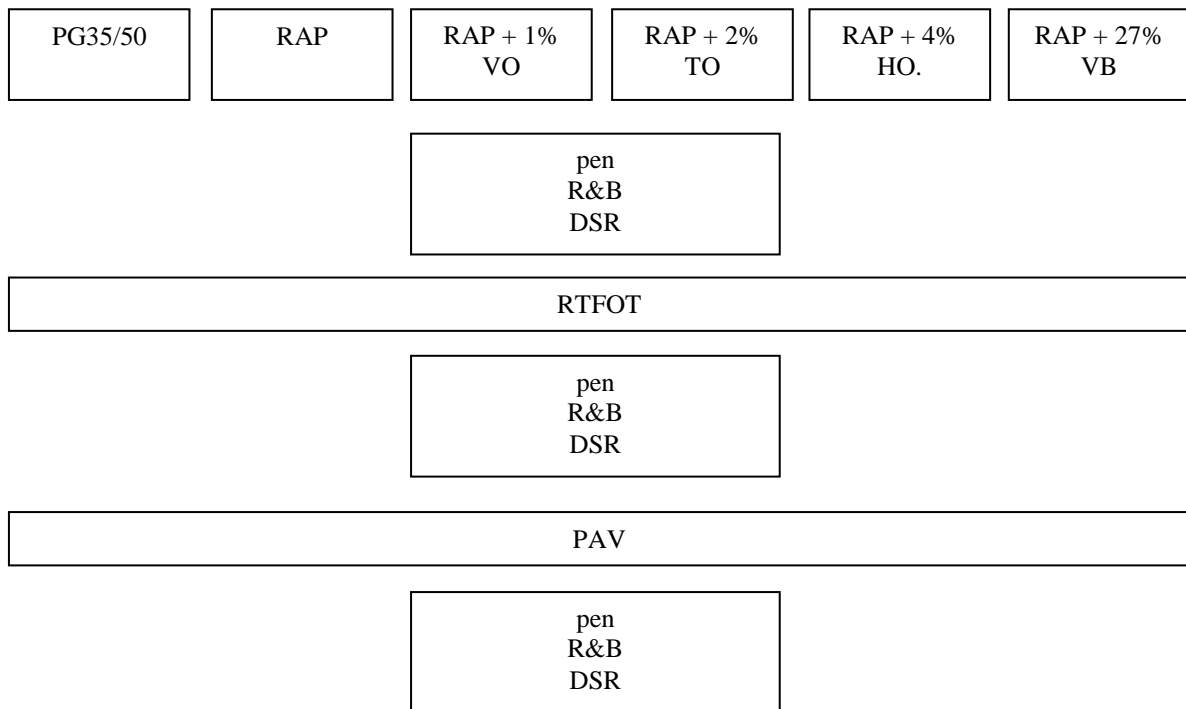
From these results, following formulations were chosen:

RAP + 1% vegetable oil VO

RAP + 2% tall oil TO

RAP + 4% hydrocarbon oil HO

In addition, a pure fresh binder with a pen of 43dmm was tested, as well as a blend of RAP with a softer fresh binder ( pen 180dmm ).To obtain the desired pen of 43 dmm, 27% of bitumen with pen 180 dmm was added.



**Figure 1: Test schedule**

All testing was done according to the relevant EN norms, valid at the time of testing.

**Recovery of binder from RAP material**

EN 12697-3: Rotary evaporator with dichloromethane

**Determination of needle penetration**

EN 1426: 25 °C, 100g, 5s

**Determination of the softening point ring and ball**

EN 1427: water

**Short time ageing**

EN 12607-1: Determination of the resistance to hardening under the influence of heat and air: Rolling Thin Film Oven Test: 75min, 163°C

**Long term ageing**

EN 14769: Accelerated long term ageing conditioning by a Pressure Ageing Vessel (PAV) 20h, 90°C

**Dynamic shear rheometer**

EN 14770: Determination of complex shear modulus and phase angle – Dynamic Shear Rheometer (DSR)

Conditions:

strain : 0.01

frequencies : 0.01 Hz, 0.0215 Hz, 0.0464 Hz, 0.1 Hz, 0.215 Hz, 0.464 Hz, 1 Hz, 2.15Hz, 4.64 Hz, 10 Hz.

temperatures : 10°C, 20°C, 30°C and 40°C on 8 mm plate

40°C, 50°C, 60°C and 70°C on 25 mm plate

Blending of the additives was done in a way to simulate addition in an asphaltplant with parallel drum for RAP. This means that the recovered binder was heated up to 120°C and the additives were then added. The blend was then further heated to 160°C while being stirred.

### 3. RESULTS AND DISCUSSION

The RAP is collected from a road in the Netherlands. The age is assumed to be over 15 years. It was a DAC 0/16 surface layer with a 70/100 pen grade bitumen.

The RAP was recovered via extraction and centrifuge with dichloromethane as solvent.

The recovered binder had a penetration of 25 dmm and a softening point of 56.2°C. Sufficient amount of binder was recovered at once, to do all binder testing and prepare all rejuvenated binders.

Initial observation indicated that most additives act differently with fresh bitumen than with aged or recovered binder.

In order to ascertain the amounts that were needed for each different additive, some try outs were done with fresh bitumen with pen 25. (see table 1)

When adding the same amounts to the recovered binder, it was observed that some additives had not the same effect on fresh binder than on recovered binder. (see further in table 2: pen and R&B results)

This was clearly noticeable with the vegetable oil and to some extent the hydrocarbon oil. This means that the behaviour from the additives can not always be predicted. In case of adding virgin binder, one can simply follow the pen-rule, which is common practice. The addition of other additives needs to be tested on lab scale on the recuperated binder. One needs at least guidelines from the suppliers. One can ask to what extent it is feasible for asphalt plants to each time make lab blends to know the final properties of the binder.

**Table 2: Differences between additives with fresh and recovered RAP binder**

Pen dmm	pure	+1% VO	+2% TO	+ 4% HO
Fresh	27	43	46	42
RAP	25	33	44	39
R&B °C				
Fresh	56.8	55.0	53.6	54.2
RAP	56.2	54.4	51.5	53.6

The different blended binders were first tested on the conventional properties penetration at 25°C (EN1426) and Softening point R&B (EN1427).

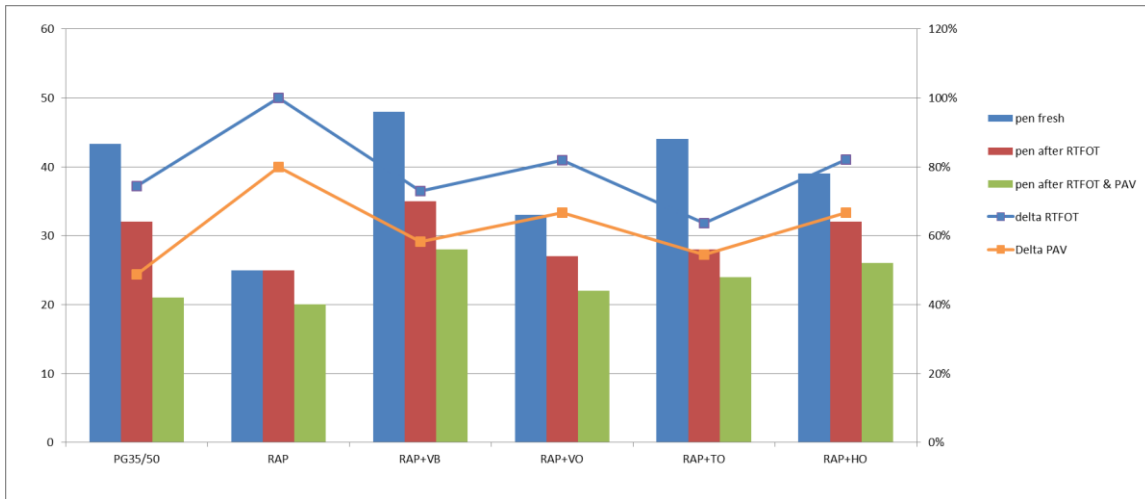
Results are summarized hereafter:

**Table 3: Penetration on binders before and after ageing**

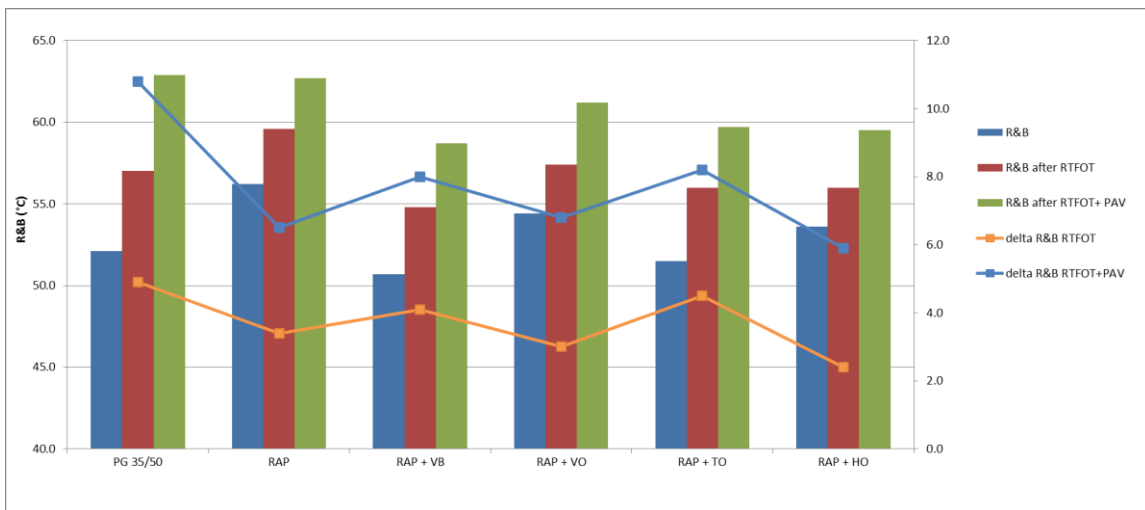
	PG 35/50	RAP	RAP+VB	RAP+VO	RAP+TO	RAP+HO
pen fresh	43	25	48	33	44	39
pen after RTFOT	32	25	35	27	28	32
pen after RTFOT & PAV	21	20	28	22	24	26
retained pen after RTFOT	74%	100%	73%	82%	64%	82%
retained pen after PAV	49%	80%	58%	67%	55%	67%
Effect on RAP by adding...	Fresh		23	8	19	14
	RTFOT		10	2	3	7
	PAV		8	2	4	6

**Table 4: Softening point of binders before and after ageing**

	PG 35/50	RAP	RAP+VB	RAP+VO	RAP+TO	RAP+HO
R&B fresh	52.1	56.2	50.7	54.4	51.5	53.6
R&B after RTFOT	57.0	59.6	54.8	57.4	56	56
R&B after RTFOT+ PAV	62.9	62.7	58.7	61.2	59.7	59.5
delta R&B RTFOT	4.9	3.4	4.1	3	4.5	2.4
delta R&B RTFOT+PAV	10.8	6.5	8	6.8	8.2	5.9
Effect on RAP by adding...	Fresh		-5.5	-1.8	-4.7	-2.6
	RTFOT		-4.8	-2.2	-3.6	-3.6
	PAV		-4.0	-1.5	-3.0	-3.2



**Figure 2: Penetration on binders before and after ageing**



**Figure 3: Softening point of binders before and after ageing**

The addition of the different additives to the RAP has a different effect on the classical tests.

- When adding fresh binder 190 pen to the RAP binder, we can observe that the rejuvenation succeeded, even somewhat more than foreseen (target pen was 43; result is 48). Also after RTFOT and PAV, values are good, and very much comparable to the pure virgin binder. The delta R&B after RTFOT and PAV are within market average for paving grade.
- When adding the vegetable oil in 1% to the RAP binder, the effect is not as expected. Increase in penetration and decrease in softening point R&B are almost neglectable. Also after RTFOT and PAV almost no difference with pure RAP binder. The changes in e.g. R&B are as small as the repeatability value for R&B. Rejuvenation effect not noticed thus with addition of 1%. A following step could be the evaluation of the addition of 2% Vegetable oil.

- When adding the 2% tall oil to the RAP binder; we obtain a good rejuvenated binder: penetration and softening point are on the same level as a fresh 35/50. However, after the first ageing step (RTFOT), part of the rejuvenation seems lost. Decrease in pen is quite high. The second ageing step is on a normal level again.
- When adding the hydrocarbon oil in 4%, we definitely see the rejuvenation effect. However, the addition of 5% might have been better or closer to the target. After RTFOT and PAV, we still observe the effect of the hydrocarbon oil addition or rejuvenation.

Besides the effect on penetration and softening point, which are very basic, we also tried to look at the changes in rheological properties.

DSR temperature and frequency sweeps were performed.

For interpretation reasons and ‘simplicity’ reasons, we picked some curves to discuss.

A first simple check was an ageing index.

Values at respectively 20°C and 50 °C at 1 Hz are taken, ratio is calculated between fresh/RTFOT and fresh/PAV.

Results are summarised hereafter in table 5.

**Table 5: Ageing index**

	20°C - 1 Hz	20°C - 1 Hz	50°C - 1 Hz	50°C - 1 Hz	52°C - 10rad/s (1.6 Hz)	52°C - 10rad/s (1.6 Hz)
	RTFOT	PAV	RTFOT	PAV	RTFOT	PAV
PG 35/50	1.5	2.7	2.0	4.6	1.9	4.4
RAP	1.0	1.5	1.2	2.0	1.2	2.0
RAP+VB	1.6	2.2	1.9	3.7	1.9	3.5
RAP+VO	1.3	1.7	1.5	2.8	1.5	2.7
RAP+TO	1.6	1.6	2.0	3.5	1.9	3.3
RAP+HO	1.1	1.6	1.5	2.4	1.4	2.3

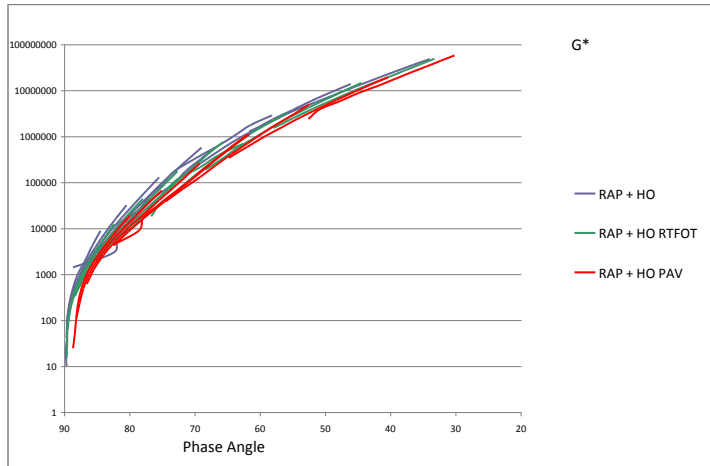
RTFOT has little effect on old binder, however PAV increases the ageing effect. Indicating the binder was not ‘end of life’.

When comparing the additives, the RAP + virgin binder acts very similar to a fresh PG 35/50 binder. The addition of tall oil also acts quite similar to RAP + virgin binder, except maybe at 20°C/PAV.



Black curves were made for all blends: fresh, after RTFOT and after PAV.

An example of RAP + HO can be found in figure 1.



**Figure 4: Black curve RAP+HO**

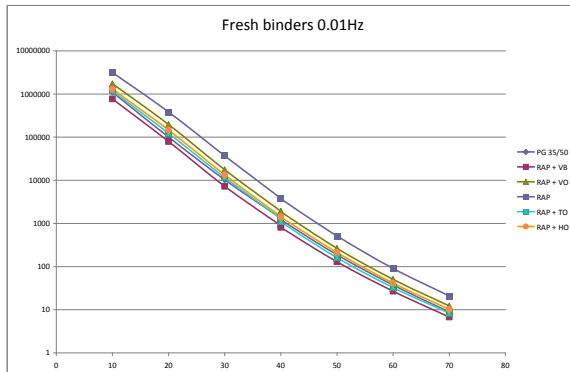
We can see that the binder is getting stiffer and less viscous with ageing, so becomes more brittle, which is normal bituminous binder behaviour.

However, to evaluate the different effects, these curves were not really suitable.

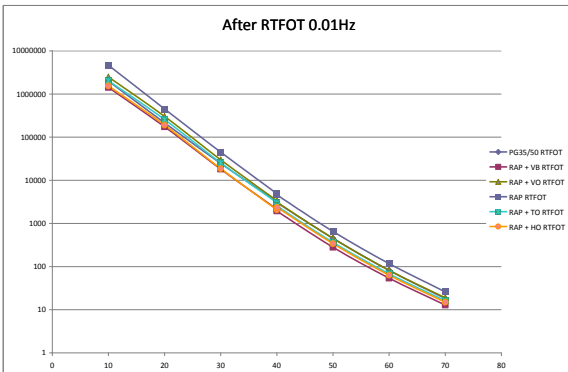
To get a clearer view, we zoomed in on chosen frequencies for each blend after each step of ageing.

In the next graphs, data is plotted for 0.01 Hz, 1Hz and 10Hz. This has been done for fresh blends, after RTFOT and after PAV aged blends.

Examples are shown for the fresh binders at the different frequencies in figure 5 to 8, also one after RTFOT.



**Figure 5: Fresh binders 0.01Hz**



**Figure 6: Binders after RTFOT 0.01Hz**

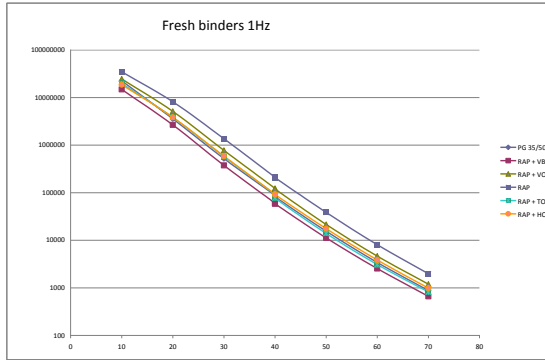


Figure 7 : Fresh binders 1Hz

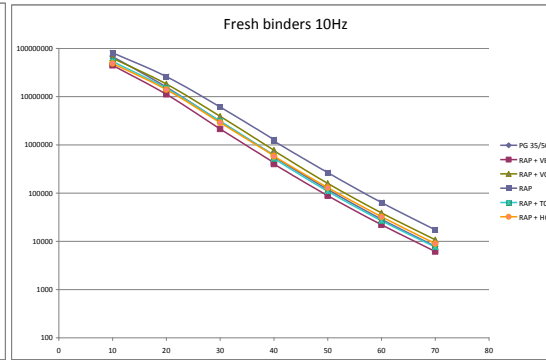


Figure 8 : Fresh binders 10Hz

It is observed that for all three steps, from fresh to short and long term ageing, the biggest differences are seen at the lowest frequency.

Looking at the figure 5, 7 or 8 for the fresh blended binders, it is observed that adding virgin binder (VB) to the RAP seems to have the most effect on the decrease of the stiffness at all temperatures and frequencies tested. This confirms also the findings of the penetration test. Adding virgin binders is much more effective than adding vegetable oil. Especially at lower frequencies, the addition of adding vegetable oil is not bringing added value.

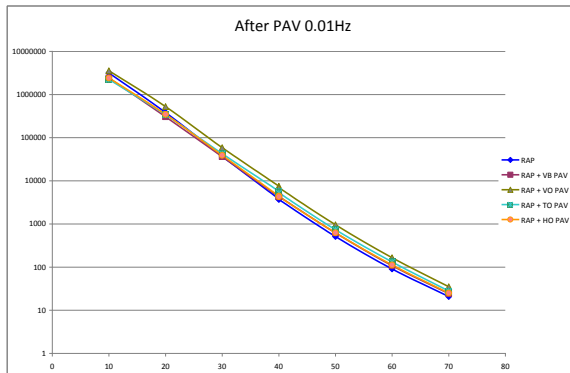


Figure 9 : PAV aged 0.01Hz

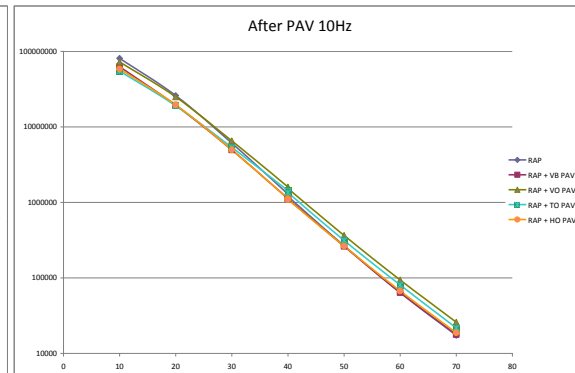


Figure 10 : PAV aged 10Hz

When looking at the RAP rejuvenated with virgin binder after ageing, (figures 9 and 10), we end up with the same rheological behaviour as the RAP in the beginning of the experiment. The rejuvenated binder has aged to the same level as the original RAP. In other words, this can be seen as successful rejuvenation as the result is the same as after the first 15 years of ageing.

The others additions seem to have led to somewhat minor results. Ranking the additives in order of beneficial effects, we come to this ranking: virgin binder, hydrocarbon originated oil, tall oil and vegetable oil. For the rejuvenation with hydrocarbon oil, nearly the same effects as the addition with virgin bitumen are observed. This might be optimised by adding a bit more hydrocarbon oil to the RAP, e.g. 5% (as the RAP binder did not react exactly the same way as virgin binder when adding 4%, see before). This is something to investigate in a next step.

The addition of the vegetable oil has the less effect. However, also here we noticed that the vegetable oil acted different with the RAP binder than with virgin material, and that the addition of 1% seems not enough. So it could be worthwhile to investigate the addition of 2 or 3 % of vegetable oil and check if the rejuvenation can be improved.

These observations were noticed in the same way for all frequencies tested.

Theoretically, one could conclude that one can perpetually rejuvenate aged binders by adding virgin binder and always end up with the same properties as after the initial ageing of completely new asphalt.

#### **4. CONCLUSIONS**

In the first part of the project, described in this paper, following observations could be made:

- There can be a difference in results in adding rejuvenating additives to a fresh binder compared to a RAP binder.
- The effect is not the same for all rejuvenating additives.
- The best effect is seen with the most common used technique, adding fresh (soft) binder to the RAP binder.
- Hydrocarbon oil and tall oil also show rejuvenating effect, but not yet at the same level as the virgin binder addition.
- The vegetable oil is not that effective; however rejuvenating effects can be noticed and might be optimised by increasing the amount of vegetable oil.
- All effects are best noticeable at lower frequencies.
- Long term ageing (PAV) shows that some blends continue to age or oxidize further than the original RAP in our study or the RAP rejuvenated with virgin binder.

These findings were an excellent starting point for further investigations.

Following items need further exploration on binder level:

- The addition of a higher amount of vegetable oil and hydrocarbon oil
- Why does a RAP binder act different than a virgin binder?
- What causes the good rejuvenation effect of a virgin binder versus the less effect of the vegetable oil? Is it related to the molecules present and the compatibility between bitumen and additive?
- Rheological behaviour: checking the change in visco-elastic behaviour; calculating SHRP parameters for e.g. rutting, and compare.

Of course the findings on these binder tests need to be confirmed in asphalt testing. The idea is to make asphalt with the same RAP material, add the rejuvenating additives to the mix in the same amount and test further in the standard functional requirement tests such as rutting, watersensitivity, stiffness and fatigue.

We look forward to report this in a next E&E conference.

#### **REFERENCES**

- [1] The Role of an Asphalt Rejuvenator in Pavement Preservation: Use and Need for Asphalt Rejuvenation, Jim Brownridge, Tricor Refining, LLC, Bakersfield, California, United States; First International Conference on Pavement Preservation; 2010
- [2] Rheological aspects of the rejuvenation of aged bitumen, Ramón Romera, Anton Santamaría et al; Rheol Acta (2006) 45: 474–478 ; DOI 10.1007/s00397-005-0078-7
- [3] Nynas, internal reports and minutes, Enhanced recycling project 2010 - 2011.