Experimental Evaluation of the Potential of the Sap of the Petroleum Plant as an Asphalt Binder Rejuvenating Agent

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ABSTRACT

The recycle of asphalt materials is increasing every year on paving industry, mainly for economical reason, once it reduces the consumption of raw-materials such as aggregates and asphalt binders. The use of higher contents of RAP is a challenge, since that reclaimed asphalts are typically aged and show brittle behavior, requiring the use of binder rejuvenators. The present work aims to investigate the potential of the sap of the Petroleum Plant (*Euphorbia Tirucalli*) as an asphalt rejuvenating agent. For this purpose, a base asphalt binder PG 64-22 was previously short and long term aged (by RTFOT and PAV, respectively), and then mixed with the sap in the content of 30%, by weight. All the samples (Original, RTFOT aged, PAV aged, and 30% sap added) were submitted to physical tests (penetration and softening point), rheological tests (Rotational Viscosity and Frequency Sweep on DSR) and MSCR test. The results showed that, with the addition of the sap, penetration, softening point, master curves, rotational viscosity and MSCR parameters of the PAV aged sample returned to their conditions right after RTFOT procedure, meaning that the sap was able to superimpose the effects of the oxidative aging. Thus, the sap of the Petroleum plant showed potential as an asphalt rejuvenator.

Keywords: Petroleum Plant; Asphalt Rejuvenating Agent; Reclaimed Asphalt Pavement; *Euphorbia Tirucalli*.

1. INTRODUCTION

The increasing use of recycled asphalts in pavement layers has been a response to the demand for more sustainable practices in the paving industry, in order to reduce the consumption of asphalt binders and aggregates in the construction of new layers. The recycling consists on adding a percentage of RAP (Reclaimed Asphalt Pavement) to a new asphalt mixture, so that the characteristics of the virgin asphalt binder can counteract the rigid and brittle behavior of the aged RAP.

The major challenge on asphalt pavement recycling has been to use increasing amounts of RAP to the new mixtures. For this, it is necessary to employ additives that can reduce the effects of oxidative aging on reclaimed asphalts. These materials are known as rejuvenating agents.

Commercially available rejuvenating agents are usually petroleum-derived additives or contain petroleum derivatives, and thus have considerable toxicity. Therefore, the search for rejuvenating agents from renewable sources that cause less damage to worker's health and the environment has also been encouraged.

The rejuvenation of cold recycled asphalt with vegetable oils as rejuvenating agents (a commercial rejuvenating vegetable oil, a grape seed oil, a flaxseed oil and a waste cooking oil) was studied in laboratory and in field tests [1]. The recycled asphalts showed to be susceptible to action of water and curing time. At the end of the study, the authors concluded that vegetable oils were suitable for the reactivation of RAP characteristics.

The use of waste cooking oil as a rejuvenating agent inspired the development of many other studies [2-4]. This material was able to superimpose the effects of oxidative hardening on the empirical properties and viscosity of a base binder over several stages of ageing, as well as to reduce the asphaltene/maltens ratio of the aged samples [2]. It was observed by [3] that there is an optimum oil content (which is different for each binder sample) which is capable of recovering the complex modulus, phase angle and the permanent deformation Superpave parameter of the aged binders.

The influence of the waste cooking oil and a commercial rejuvenating agent on the formation of clusters of RAP on recycled asphalts was also investigated, and it was observed that the addition of the rejuvenators increased the clusters content [4]. The authors concluded that the formation of clusters is undesirable since they can promote heterogeneity of the mixtures and may prevent uniform distribution of the virgin binder.

Another material under investigation for recycling purposes is an extract from the Guayule plant (*Parthenium Argentatum),* a woody shrub which grows in arid and semi-arid climates and is a source of high quality natural rubber [5]. This extract was compared to a commercial rejuvenating agent known as *Cyclogem L.* The results indicated that recycled mixtures with the alternative material were more resistant to rutting, however more susceptible to moisture damage and thermal cracking than the commercial product. Nevertheless, the authors concluded that the Guayule extract presented great potential as a renewable source of alternative binder or asphalt additive.

The Petroleum Plant (*Euphorbia Tirucalli)* is shrub commonly found in dry regions of Brazil, whose sap has viscous and adhesive properties. This sap has been investigated as an asphalt modifier, and has shown solvency and anti-aging effects when added in the asphalt binder [6]. Thus, its properties indicate that this material can be a candidate to be employed as rejuvenating agents of RAP. The present work aims to investigate the potential of the sap of the Petroleum Plant for application as an asphalt rejuvenator for application on recycled asphalt layers.

2. MATERIALS AND TESTING METHODS

In this research, an asphalt binder classified as PG 64-22 was short-term aged on rolling thin film oven (RTFO) according to ASTM D2872 [7], in order to simulate the oxidative aging due to mixing and compaction procedures. The RTFO aged binder was then submitted to long-term aging in pressure vessel (PAV) following ASTM D6521 [8] recommendations.

Afterwards, 30% of the sap of *Euphorbia Tirucalli* (Petroleum Plant) was added to the aged base binder in a low shear mixing reactor at 140ºC, 1500 rpm for 60 minutes. This new material is designated as 30%. The sap was previously oven dried and had the form of lumps by the moment of application.

All samples (unaged, post-RTFOT, post-PAV and 30%) were subjected to the following tests:

- Empirical characterization by Penetration Test (ASTM D5) [9] and Softening Point Test (ASTM D36) [10];
- Rotational Viscosity, following ASTM D4402 [11], performing test at Brookfield viscosimeter, with spindle 21, 20 rpm, at 135ºC, 150ºC and 177ºC;
- Frequency Sweep Tests in Dynamic Shear Rheometer (DSR), under stress control mode, at a range of 1-160 rad/s, at high (46ºC – 88ºC) and intermediate (40ºC – 4ºC) temperatures, as recommended by ASTM D7175 [12]. The master curves were built by Time-Temperature Superposition Principle, at reference temperature of 25ºC.

- Multiple Stress Creep and Recovery Test, according ASTM D7405 [13].

3. RESULTS AND DISCUSSION

3.1 Empirical Properties

The penetration values and the softening points of the unaged, aged and rejuvenated samples are shown in Figures 1 and 2, respectively.

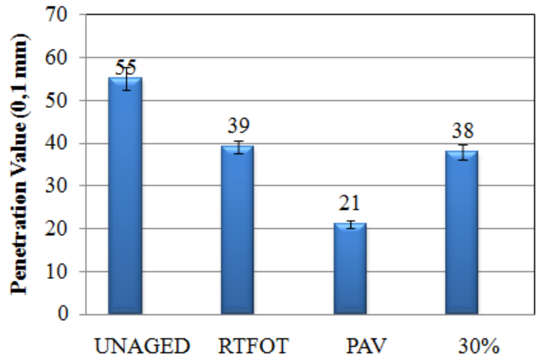

FIGURE 1 Penetration values of the unaged, aged and rejuvenated samples

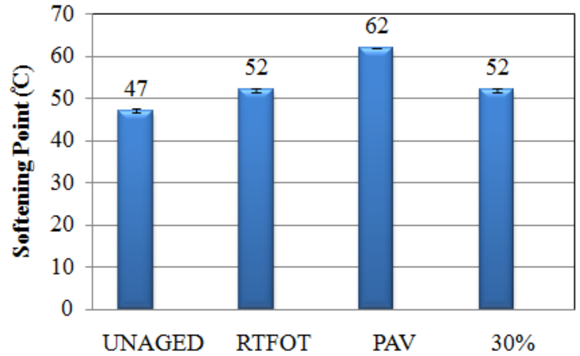

FIGURE 2 Softening Point of the unaged, aged and rejuvenated samples

The aging process reduced the penetration value and increased the softening point of the asphalt binder, showing the hardening of the binder sample due to oxidative and volatile loss processes. The addition of the sap to the aged material, however, increased its penetration and reduced the softening point, returning them to the values observed right after aging in RTFOT. This indicates that the addition of sap was able to undo the long-term aging effects previously experienced by the binder sample on the empirical properties. These results are equivalent to the effects of the addition of waste cooking oil to binders subjected to simulation of short and long term aging: with the addition of increasing oil contents it was observed an enlarge in penetration and a reduction in the softening point [3].

3.2 Rotational Viscosity

The viscosity-temperature chart of the samples are shown in Figure 3 and the Table 1 presents the viscosity increments of each sample regarding the unaged asphalt binder, for all test temperatures.

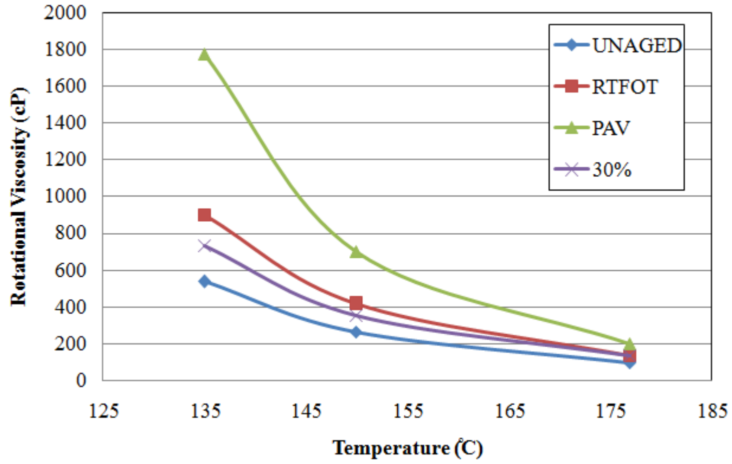

FIGURE 1 Rotational Viscosity of the unaged, aged and rejuvenated samples

TABLE 1 Viscosity increments of the base binder due to aging and rejuvenating processes

Viscosity Increments			
Temperature	**RTFOT**	**PAV**	**30%**
135ºC	68,1%	230,3%	36,6%
150ºC	56,9%	164,7%	34,2%
177ºC	40,1%	110,4%	43,8%

The viscosities presented by the rejuvenated samples were even lower than the viscosities observed after short-term aging, except at 177ºC. The viscosity increments for the rejuvenated samples were approximately half of the ones observed for RTFOT aged samples, showing that the addition of sap recovered not only to the effects of long-term aging, but half of the effects of short-term aging as well.

The addition of sap had more visible effects at lower temperatures, where the increments in viscosity due aging were stronger. At 177 ° C, the sap rejuvenated sample had a viscosity slightly higher than the short-term aged sample, contrary to all the results observed so far. This trend discrepancy can be explained by the thermal behavior of the sap, which overgoes a first order transition around 177ºC [6].

The effects of adding other two rejuvenating agents (among them the waste cooking oil) on the viscosity of a binder recovered from recycled asphalt were also temperature dependent [4]. Moreover, these authors found out that the effects of rejuvenation are greater at lower temperatures (135°C) than at higher temperatures (175°C), where the recovered binder exhibits practically the same viscosity as the rejuvenated binders.

3.3 Rheological Parameters

The master curves of the complex modulus and phase angle are presented at Figures 4 and 5, in this order.

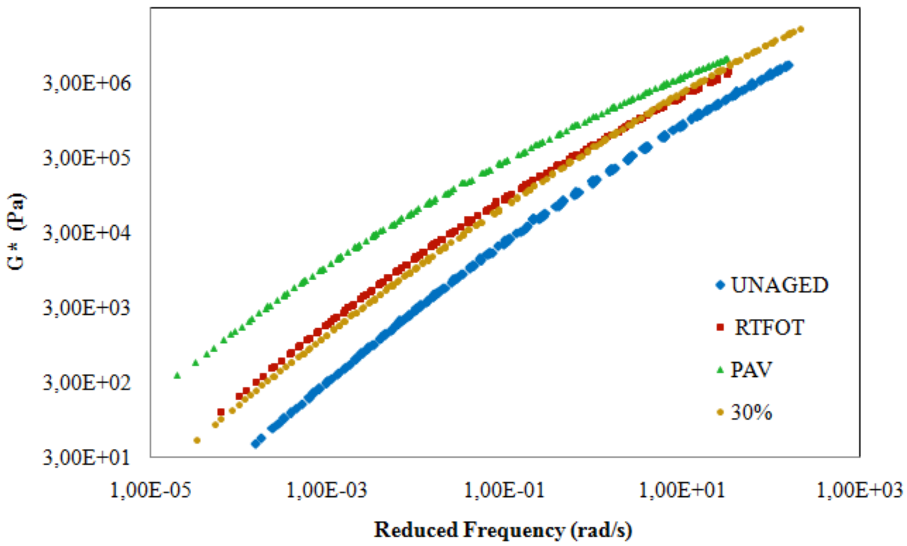

FIGURE 4 Master Curves of G* of unaged, aged and rejuvenated samples

As expected, oxidative aging processes increased the complex modulus and reduced phase angle of the reference binder, and this effect is more pronounced after long-term aging. After the addition of the sap, the complex modulus reduces, returning at levels similar to those obtained after RTFOT. These results are equivalent to those observed on [3], where two rejuvenating agents (commercial sample and waste cooking oil) significantly reduced the complex modulus of a reclaimed asphalt.

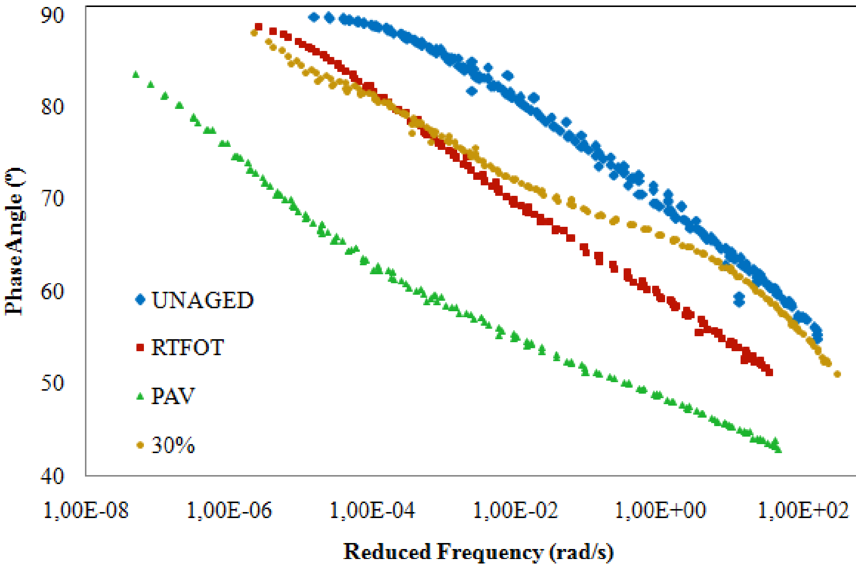

FIGURE 5 Master Curves of δ of unaged, aged and rejuvenated samples

Regarding the phase angle, the curve of the rejuvenated sample presented the formation of a plateau and moved towards higher values of δ, when compared to the PAV aged one. The formation of the plateau at the center of the curve caused the phase angle to reach values close to those observed by the RTFOT sample at low frequencies (high temperatures), and close to the ones observed for the unaged sample at high frequencies (intermediate temperatures). This behavior indicates improvements on the elastic behavior of the material at elevated temperatures (where the greatest concern is permanent deformation) and a higher ability to flow at low temperatures (where fatigue has more important effects), within the linear viscoelasticity zone.

3.4 Multiple Stress Creep and Recovery

The parameters of the MSCR tests for all the aging conditions are shown in Table 2.

TABLE 2 MSCR Parameters of the unaged, aged and rejuvenated samples

Sample	R100 (%)	R3200 (%)	Jnr 100 (kPa^{-1})	Jnr 3200 (kPa^{-1})	Rdiff (%)	Jnrdiff (%)
UNAGED	8,1	2,0	2,116	2,516	75,1	18,9
RTFOT	14,0	5,9	0,859	1,014	57,9	18,0
PAV	41,3	33,6	0,133	0,153	18,8	15,4
30%	31,6	5,6	1,013	1,797	82,4	77,4

At lower stress, it was observed that the addition of sap reduced the percent recovery of the PAV aged sample, but it almost doubled the value observed after RTFOT aging, showing more elastic behavior. At high stress, however, the addition of the sap resulted in a percent recovery similar the RTFO aged sample, showing that the rejuvenated material showed a greater susceptibility to the stress variations. This observation was confirmed by the parameter Rdiff, which was much higher for the rejuvenated condition than for the other conditions.

Regarding the non-recoverable creep compliance, it was verified that the addition of the sap caused the material to present close but higher values than the short-term aged sample, showing that the rejuvenated sample is slightly more susceptible to permanent deformation than the RTFO sample, although the values found classify them as fit to the same level of traffic (V - very high).

These results are even more promising than those observed with waste cooking oils as rejuvenating agents. When analyzing the resistance to permanent deformation of rejuvenated asphalt samples with these materials, according to the Superpave parameter, it was found that the rejuvenated samples presented a poor behavior in relation to the permanent deformation, presenting lower reference temperatures with the increase of the additive content [3].

4. CONCLUSIONS

The addition of the sap of the Petroleum Plant to the long-term aged asphalt binder sample has, in general, restored almost all the characteristics to their conditions right after the short-term aging. It was observed that the sap reduced the effects of hardening due to oxidation and loss of volatiles: increased penetration, reduced softening point, lowered rotational viscosity, reduced complex modulus, increased phase angle and decreased non-recoverable creep compliance.

This variation not only in the magnitude of the effects but also in some trend of behaviour (as it was observed on δ Master Curve) may indicate that the action of the Petroleum Plant is not of merely solvency but involves a series of reactions that promote a better balance between the maltenic/asphaltene portions of the binders, promoting a real asphalt rejuvenation. The results indicated, therefore, that the sap has potential to be used as a rejuvenator agent to reclaimed asphalts on recycled pavements.

5. ACKNOWLEDGMENTS

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