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Study of incorporation of footwear industry residues in asphalt mixtures

Dennyele Alves Gama[1], Adriano Elísio de Figueiredo Lopes Lucena[1], Thalita Maria Ramos Porto[1], Jeovanesa Régis de Carvalho[1]

[1] Laboratório de Pavimentação - LEP – UFCG, CEP: 58429 – 900, Campina Grande, Paraíba, Brasil.
e-mail:dennyelealves@gmail.com, lucenafb@uol.com.br, thalita_porto8@hotmail.com, nesaregis@hotmail.com

ABSTRACT

The footwear industry discards residues in its most diverse phases of production, the Ethylene Vinyl Acetate (EVA) being one of them. This work proposes an evaluation of the use of EVA's residue discarded by the footwear industry of Campina Grande city in asphalt mixtures. The modified asphalt binder was characterized by penetration test, softening point, and rotational viscosity. The asphalt mixture was prepared through the SUPERPAVE methodology. The EVA's residue incorporated to Petroleum Asphalt Cement (CAP) through the wet process in percentages of 0, 2, 3, 4 and 5 % in mass. Among the asphalt mixtures analyzed, the mixtures that contain 2% of EVA residue and 3 hours of aging were the most important, as they met the minimum criteria for Resistance to induced moisture damage (Lottman). The results show that EVA`s residues are an effective alternative for use in asphalt paving.

Keywords: industrial residue, footwear industry, asphalt mixtures, paving.

1. INTRODUCTION

On August 2nd of 2010, law nº 12.305 was sanctioned in Brazil. This law institutes a National Policy of Solid Residues, which represents a regulatory milestone for the sector. Among the main types of residues produced worldwide by industries, rubber's residue stands out for being a material hard to recycle, because of its complex and heterogeneous composition, which makes it difficult or even impossible to reuse.

However, since 1963, techniques for the reuse of tires' rubber are being developed for maintenance, restoration, and extension of pavement's useful life. These techniques improve traditional binders performance on some important characteristics regarding the mechanical behavior of asphalt mixtures [17].

Rubber coming from footwear industry are also strong sources of environmental pollution and waste of prime matter. LUO e CHEN [15] say that these materials show a relatively high resistance to biological agents and to adverse weather, consequently causing major problems to communities in general when discarded.

The polymer EVA (Ethylene Vinyl Acetate) while in mass and with vinyl acetate contents between 18% and 28% have great application in footwear industry [18]. In the production process, approximately 20% of EVA become leftovers, producing the estimated discard amount of 7,932 tons per year in Brazil, which is wasted as the residues are sent to sanitary landfills and landfills [18]. Thus, economic aspects and environmental pollution are both relevant reasons for efforts to promote the reuse and/or recycling of these polymeric materials to exist.

Due to its aliphatic nature, EVA solubilizes in the saturated fractions of the asphalt, due to the existence of ethylene sequences of high molecular weight, modifying the flow of the material [13]. [12] made use of the dry process of asphalt mixing with the EVA residue, studying the mechanical properties of modified asphalt mixtures with 0, 1, 2 and 3% EVA residue contents and with 0, 2 and 4 hours of aging. Their results showed that the use of this residue increased the resistance of the asphalt mixtures to fatigue, but the mixtures became more susceptible to permanent deformation.

Therefore, knowing the potential for paving Brazil has, the issues regarding the inadequate destination given to EVA expanded sheets leftovers, and before the development of technologies related to the reuse of rubber in asphalt paving, there was a motivation to insert EVA's residues from footwear industries as modifying agents in Petroleum Asphalt Cement, intending to provide an environmentally adequate destination for

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the leftovers and to promote a reuse alternative for this industrial residue.

2. MATERIALS AND METHODS

2.1 Used materials

2.1.1 Petroleum Asphalt Cement & aggregates

Petroleum Asphalt Cement (CAP) 50/70 was used. It was provided by the company JBR Engineering. The aggregates used in the asphalt mixture were gravel 19mm, gravel 12.5mm, sand and stone dust (Table 1). The filler used was hydrated lime.

Table 1: Physical characterization of aggregates

AGGREGATES	APPARENT SPECIFIC GRAVITY (g/cm³)	BULK SPECIFIC GRAVITY (g/cm³)	ABSORPTION (%)	LOS ANGELES ABRASION (%)
Gravel 19 mm	2,75	2,76	0,21	37,06
Gravel 12,5 mm	2,76	2,80	0,46	44,50
Sand	2,54	2,55	0,08	-
Stone dust	2,49	2,54	0,72	-

2.1.2 EVA's residue

The EVA's residue (EVAR) used in this research came from the footwear insoles making of a foot-wear factory from the city of Campina Grande - PB. The material was provided in pieces and glued to a film made of synthetic material. The residue was manually unglued from the synthetic material and cut to smaller sizes (Figure 1) to be taken to the grinder to achieve the desired size-distribution.

Figure 1: Cut EVA's residue.

EVA's residues particle size has low influence on the properties acquired by CAP through modification [10]. The chosen size-distribution was EVA's residue particles that go through sieves #10 (correspondent to 2mm).

A grinder of the brand Primotécnica model P1-003 that possesses 3 rotatory knives and 2 fixed ones with 3 different sieves for the wished size-distribution was used. The material ground was sieved so as to use only the passing particles in sieve #10 (2.00mm). The granulometry chosen was based on the study carried out by [6], which used EVA residue particles that pass through the 9 mesh sieve (corresponding to 2mm).

2.2 Methods

2.2.1 Residue's classification

For a residue to be sorted properly, it is necessary that its chemical composition is determined according to the procedures suggested by NBR 10004 [3]. This norm determines the criteria adopted for residues classification regarding their potential risks for the environment and public health.

Considering the above, and aiming to environmentally classify the EVA's residue, we used procedures for obtaining leaching extract [3] and solubilized extract [4] from solid wastes.

The samples were prepared in Lab of Environmental Management and Residues Treatment (Laboratório de Gestão Ambiental e Tratamento de Resíduos - LABGER), part of the Academical Unity of Chemical Engineering (Unidade Acadêmica de Engenharia Química - UAEQ) from Federal University of Campina Grande (Universidade Federal de Campina Grande - UFCG) and sent for analysis to Mining Support Fund (Fundo de Apoio à Mineração - FUMINERAL) in Goiás/GO. Grinded EVA passing through sieve 2.00mm was utilized, as it was for the incorporation into CAP. Asphalt Cement (CAP) 50/70 was used. It was provided by the company JBR Engineering. The aggregates used in the asphalt mixture were gravel 19mm, gravel 12.5mm, sand and stone dust. The filler used was hydrated lime.

2.2.2 Mixing CAP with EVA's residue

As data from BRASKEM [6] state, the ideal temperature to modify CAP with EVA polymer is of 150°C. This mixing temperature must not exceed 200°C to not damage the EVA's characteristics. KALANTAR et al. [13] suggest that the mixing temperature should not surpass 185°C, otherwise the CAP could oxidize. The mixing time must be enough to obtain a homogeneous mixture through the dispersion of plastic residue inside the matrices.

KALANTAR et al. [13] verified that mixtures with EVA contents below 4% had no significant changes compared to conventional binders and that mixtures with EVA content above 6% were extremely viscous, becoming inadequate to be used.

Therefore, for the production process of CAP with EVA, a FISATOM mechanical agitator (model 72) was used. The CAP was initially heated to a 160°C temperature. The EVA was added in contents of 2, 3, 4, and 5% and the mixture was agitated in 544 rotations per minute (RPM) for 2 hours after all the residue was added. The components were mixed with frequent agitation, as we kept attentive so it would not exceed the reaction's temperature and time.

Tests for physical characterization of the CAP samples were performed in UFCG's lab intending to verify the properties both of pure CAP 50/70 and of CAP modified with different contents of EVA's residue. 3 specimens were prepared for each content of EVA. The norms adopted for performing these tests are presented in Table 2.

Table 2: Norms for physical characterization tests of CAP samples.

TESTS	CAP 50/70	LIMITS	METHODS
Rotational viscosity 135ºC (cp)	546,50	274 (min.)	NBR 15184/2007
Rotational viscosity 150ºC (cp)	266,50	112 (min.)	NBR 15184/2007
Rotational viscosity 177ºC (cp)	95,25	57 – 285	NBR 15184/2007
Softening point (ºC)	44,55	46 (min)	NBR 6560/2000
Penetration (0,1mm)	59,5	50-70	DNIT ME 155/2010

2.2.3 Asphalt mixture

For the asphalt mixture preparation, the SUPERPAVE (Superior Performance Asphalt Pavements) methodology was utilized. This methodology's objective is to develop an economic mixture of asphalt binder and aggregate that reaches a compatible performance level with traffic demands and pavement structure.

The first step of the SUPERPAVE methodology comprised the choice of three granulometric compositions with available aggregates (gravel 19 mm, gravel 12.5 mm, sand, stone dust, and filler), within the C Range of DNIT granulometry, with a maximum nominal size of 19mm. The upper, intermediate and lower granulometric curves contain the proportions of aggregates shown in Table 3.

Table 3: Particle size distribution

	GRAVEL 19 (%)	GRAVEL 12 (%)	SAND (%)	STONE DUST (%)	FILLER (%)
Upper	6	35	14	43	2
Intermediate	15	35	6	42	2
Lower	26	34	0	40	0

After the determination of the granulometric compositions of the three mixtures (lower, intermediate and upper) and from the proportions of aggregates associated to the physical characteristics of the aggregates, the initial binder content was obtained. The test specimens can be compressed into different spinning numbers according to the volume of traffic that was considered. In this research, traffic was considered Medium to High, thus Nbegin = 8 turns, Nproject = 100 turns (in which air voids - Va - must be equal to 4%) and Nmaximum = 160 turns in the SUPERPAVE rotary compactor. The Nbegin and Nmax are only used to evaluate the compactability of the asphalt mixture, and the Nproject must meet 4% of Va and is used to select the binder content of the mixture.

The compaction was performed on the SUPERPAVE gyratory compactor (Figure 2) with an applied pressure of 600kPa and the rotation angle of 1.25 ° according to the SUPERPAVE methodology. For the initial binder content, the value of 4.9% was calculated and 18 specimens were molded to check the air voids obtained, with 6 specimens for each particle size curve divided into two specimens for Nb, two for Np and two for Nm. From the volumetric parameters presented in table 4, it was possible to choose the most satisfactory particle size distribution, which should have air voids of 4%. Therefore, the upper curve was the one that obtained air voids closer to that established by the methodology criteria.

Figure 2: SUPERPAVE gyratory compactor.

Table 4: Volumetric parameters of asphalt mixtures.

	CAP %	%Gmm Nbegin	%Gmm Nproject	%Gmm Nmaximum	VMA (%)	Va(%)
Upper	4,9	91,87	97,41	97,55	12,48	2,6
Intermediate	4,9	90,78	96,77	97,35	12,13	3,2
Lower	4,9	88,68	96,29	96,18	13,48	3,7
Methodology criteria	-	<89%	96%	<98%	13 min	4

If the air voids of 4% with the initial CAP content of 4.9% was not found, an estimate of the binder content would reach 4% of Va. The curve that fit the assumptions of % VMA (voids in mineral aggregates), and % Gmm (maximum specific gravity) was the Upper curve. Therefore, the other compaction steps were performed using the particle size distribution of the upper curve. The new calculated CAP content was 4.8%.

Thus, other three contents are considered, in addition to the estimated content: estimated content ± 0.5% and + 1% from where 6 additional specimens are molded for each content. In this stage, it was not possible to find the air voids of 4%, so, from the curve of CAP content as a function of the air voids (Figure 3), a new CAP content of 5.06% was this criterion, and again six test pieces, this time all in the Np with 100 turns, were molded for the verification of the volumetric properties of the compacted mixture and confirmation of the value of 4% of air voids.

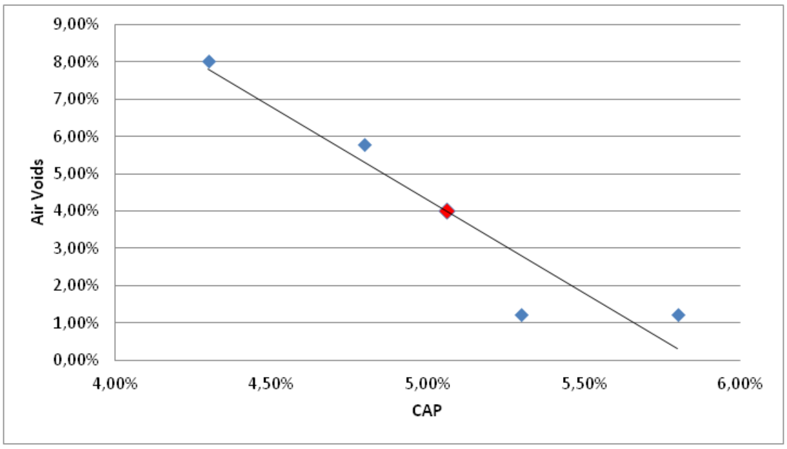

Figure 3: Estimation of the optimal content of CAP

The specimens compaction for posterior mechanical properties evaluation was made in a gyratory compactor. The asphalt mixture stays in a greenhouse for a 2 hours period before the compaction, in order to simulate short-term aging that happens during the machining. In this study, different times of aging beyond the 2 hours were used, aiming to analyze this parameter's effect upon the residue variation in asphalt mixtures. 3 specimens were prepared for each content of EVA.

The tests made for the asphalt mixtures mechanical characterization were Resistance to Moisture Induced Damage Testing (modified Lottman) according to AASHTO's T283 [2].

3. RESULTS

3.1 Residue's classification

The metals analyzed with Leaching and Solubilization tests and the respective results are shown in Table 5.

Table 5: Analyzed metals and concentrations.

TEST	Pb (mg/l)	Mn (mg/l)	Ni (mg/l)	Zn (mg/l)	Fe (mg/l)	Cr (mg/l)	Al (mg/l)
Leaching	< 0,10	< 0,10	< 0,10	43,80	< 0,10	< 0,10	< 1,00
Limit Value	1,00	0,10	0,02	5,00	0,30	5,00	0,20
Solubilization	< 0,10	< 0,10	< 0,10	2,37	< 0,10	< 0,10	< 1,00
Limit Value	0,01	0,10	0,02	5,00	0,30	0,05	0,20

Considering the residue's classification as determined by NBR 10004 [3], we observe that the maximum limit of zinc concentration established by this norm, for a substance submitted to a leaching process, is fairly lower to the one found in the sample. For a residue to be classified as hazardous, it must contain at least one metal with contents above established. We can observe on Table 2 that the zinc contents of the EVA's residue of a footwear industry from Campina Grande are extremely high if compared to the limit established by the norm, reaching contents of 43.8mg/l, and, besides, that aluminum is also found with contents above maximum level, facts that place EVA's residue in Class I. In other words, this is a hazardous residue.

According to NBR 10004 [3], a residue's hazardousness is determined by its physical, chemical, and communicable properties, and it may be a risk to public health, by provoking mortality and incidence of diseases, or by accentuating their rates and risks to the environment, when the residue is managed inappropriately. Contact with zinc (Zn) causes damage to the human organism, such as coughing, fevers, nausea, and vomits.

Zinc is among the metals normally used as an interfacial agent for the compatibilization of elastomers and thermoplastics, such as block copolymers, as it is EVA's case. The vulcanization system starts a series of complex reactions, involving accelerators, sulfur, ZnO, and other components, with emphasis on the reaction between zinc oxide and accelerator as the main stage of the vulcanization reaction [13].

GONZALEZ et al. [11] declare that EVA's residue used to be classified as a Class II B (non-inert) residue, differently from the residue used in this research, and considered as a non-toxic and non-biodegradable material, but of low hazardousness, with possible properties such as: combustibility, biodegradability or solubility in water, and non-biodegradability. According to GONZALEZ et al [11], with this classification comes the recommendation for this residue to be landfilled or incinerated, in the latter case with risks of toxic gases liberation, for example, CO2, CO, smoke, hydrocarbons and possible traces of acrolein.

Therefore, we consider necessary a greater preoccupation regarding the inadequate discard, the treatment form, and the destination of EVA's residues to landfills for residues classified as non-hazardous wastes, once that zinc's toxicity potential may compromise the environment in which it is in, causing serious environmental problems.

By inserting EVA's residues in Petroleum Asphalt Cement, that is an impervious material, this residue could be encapsulated and thus not pollute the environment. However, studies about this perspective must be developed in order to analyze how hazardous would be to handle this material for paving application.

3.2 CAP's characterization

3.2.1 Penetration test

The penetration test determines the consistency of asphalt materials. The predicted behavior is that the addition of EVA's residue makes the binder more consistent, resulting in lower penetration levels due to the increase of EVA content in the mixtures. The obtained results are presented in Figure 4's graph.

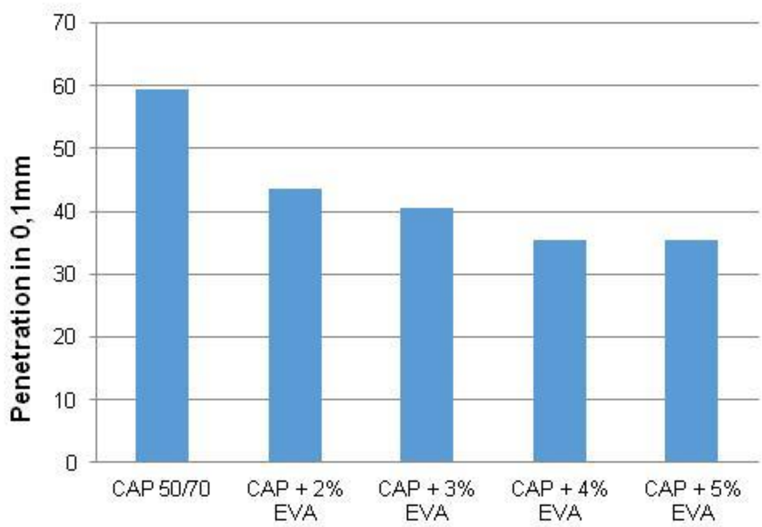

Figure 4: Penetration´s test results.

CAP in its pure state was classified according to its consistency through the penetration test in 50/70, which represents that the average penetration in the sample was between 50 and 70 tenths of millimeter. As the EVAR content incorporation into the asphalt binder was increased, it was possible to see the penetration's decrease, and consequently, the in-crease of CAP's consistency, making it harder. On 5% EVAR content level, the average penetration was of 35.5 tenths of millimeters.

Comparing this result with the ones obtained by [17], we verify a tendency of decrease in penetration in CAP face the increase of EVA content in the asphalt. SAUOLA *et al.* [17] also observed that the penetration level of CAP modified with EVAR is considerably lower if compared to CAP modified with pure EVA in the same proportions. Therefore we can affirm that EVA's residue improves CAP's consistency more significantly than the pure polymer.

3.2.2 Softening point test

The softening point is the temperature in which an asphalt binder consistency changes from plastic or semi-solid state into a liquid state. Figure 5 illustrates the effects of rubber content on the softening point of the binder modified with EVA.

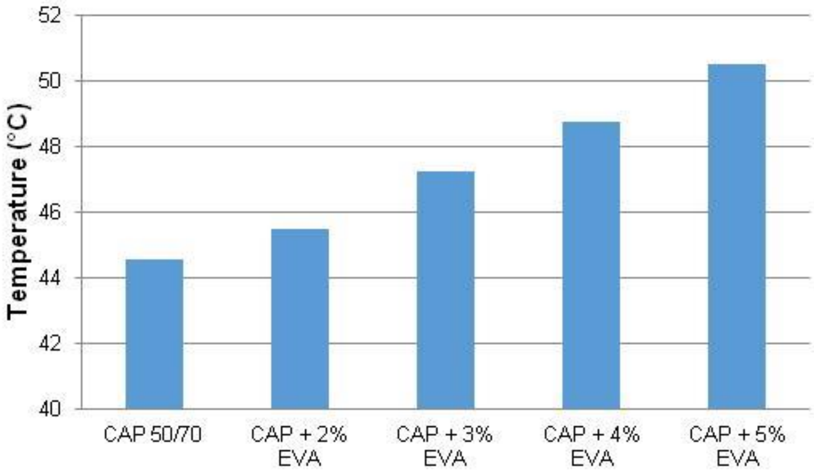

Figure 5: Softening point test results (°C).

The softening point for the pure CAP sample was of 44.5°C. We observe a gradual in-crease of temperature, proportional to the increase of EVA content, achieving 50.5°C for 5% EVA content. Thus, an increase of the binder's viscosity with an addition of EVA residue was observed, elevating the softening point, which represents an increase of resistance to accumulation of permanent deformation in modified mixtures.

This result also checks with [11], [16] and [17] studies about CAP modified with EVA, making evident the raise of modified CAP's softening temperature face the increase of EVA content.

3.2.3 Rotational viscosity

The Rotational viscosity test was performed with a spindle 21, both for pure 50/70 CAP and for modified ones. The test results for rotational viscosity with pure and modified CAP with EVA contents of 2, 3, 4 and 5% are shown in Figure 6. We can observe the increase of CAP's viscosity face the increase of EVA content. The highest viscosity shown by the modified CAP is due to the presence of copolymer residue at 5% content.

At low temperature (135°C), the addition of this polymeric residue produces a significant increase of viscosity compared to a pure CAP, as expected. Generally, these results show that binders modified with EVA have higher viscosity values compared to pure binders.

But at a temperature of 177°C, the asphalt's behavior shows no great difference between different EVAR contents, just as it shows no great difference if compared to a pure CAP. The results show that at 135°C the original binder has the lowest viscosity and the asphalt modified with 5% EVA has the highest, which is a typical characteristic of thermoplastic polymer to the binder ([18]; [10]).

EVA, due to its aliphatic nature, was solubilized in the saturated asphalt fractions, modifying the material's draining. A greater hardness was provided by the addition of EVA's residue to the asphalt, representing an accumulation of resistance to permanent deformation.

The viscosity value of the binders modified at 135°C did not surpass 3000 mPa.s [2]. This criterion is required so that the processes of pumping, handling and applying are not hampered. Thus, all binders can be utilized for paving.

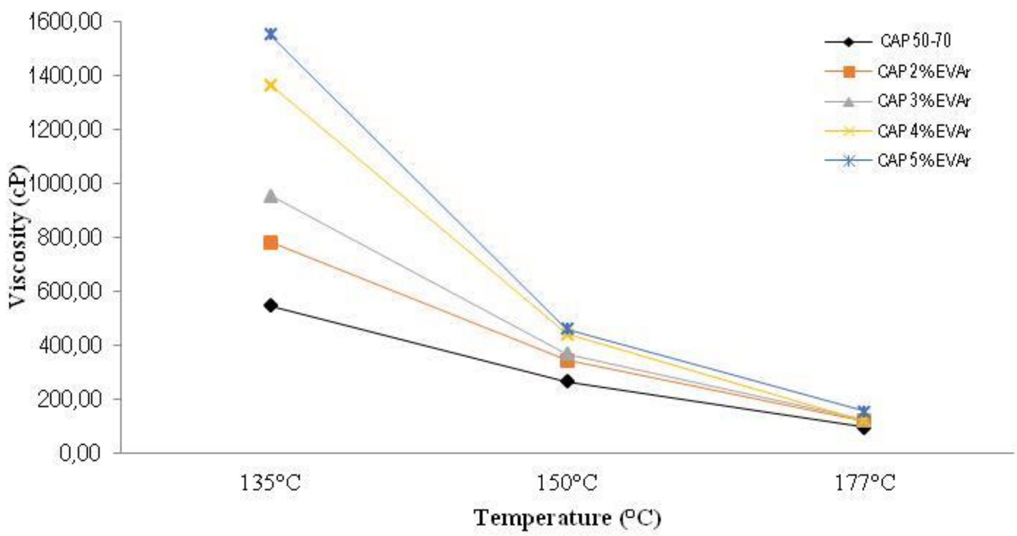

Figure 6: Viscosity of pure CAP and EVA modified CAP.

3.3 Asphalt mixture

3.3.1 Resistance to induced moisture damage (Lottman)

The analysis of resistance loss by induced moisture is made through the relation between the tensile strength of specimens with conditioning (saturation, cooling, and heating in water) and specimens without conditioning. This analysis is given by the relation (in percentage) between the conditioned specimen's TSu and the non-conditioned's TS, named Retained Tensile Strength (RTS).

In Figure 7, we can observe a decrease in the values of TSu (MPa) with increasing content of EVA's residue. The mixtures until 5% of EVAR and 5 hours of aging met the tensile strength limit of 0.65MPa, advised by DNIT's ES 031/2006. Only the mixtures with 6% of EVA's residue and 0 hours of aging and 0% of EVA's residue and 6 hours of aging were below the established limit.

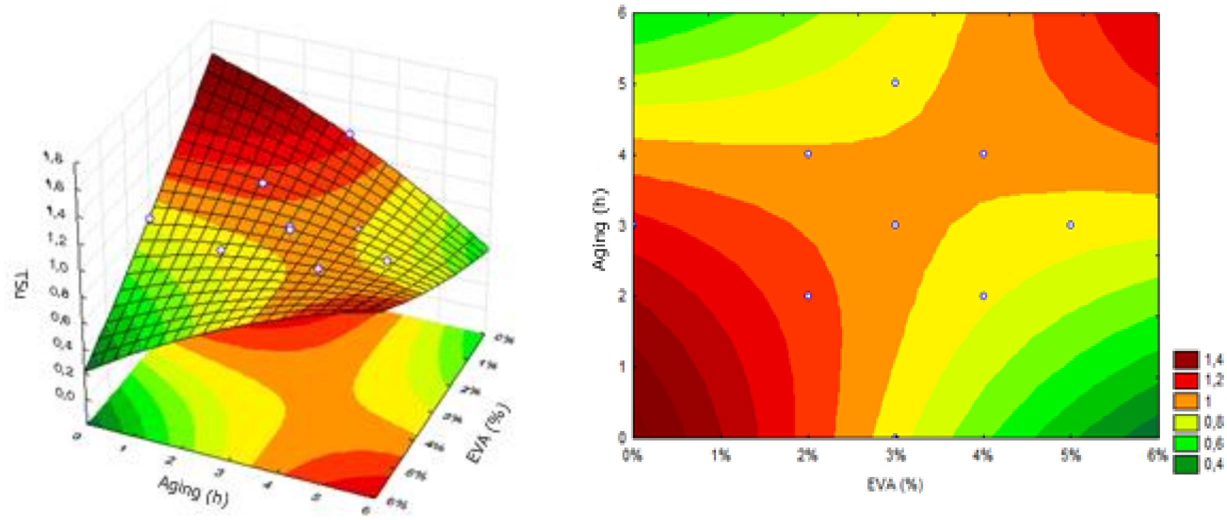

Figure 7: Response Surface for Lottman.

The Retained Tensile Strength were observed on the surface of Figure 8.

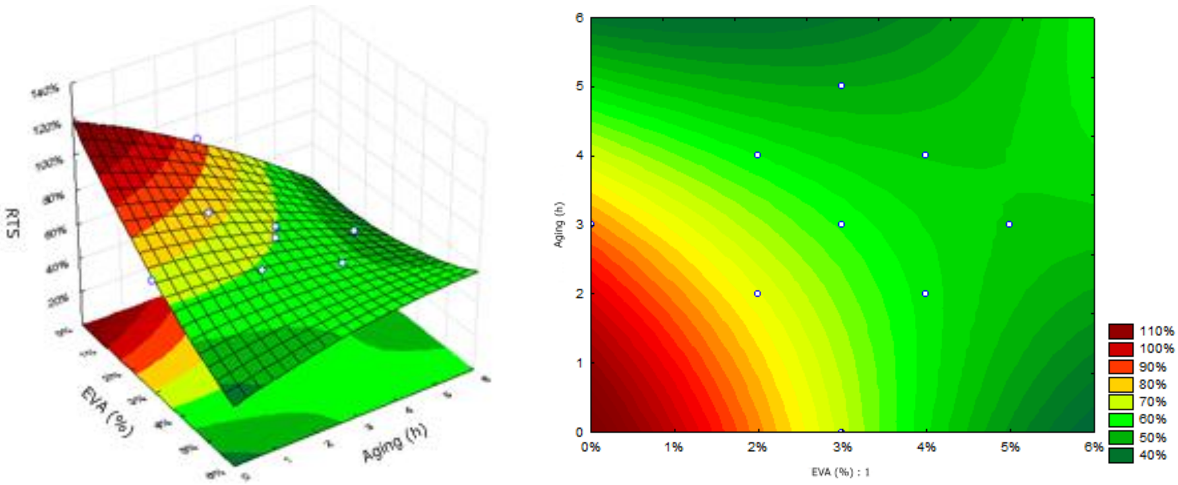

Figure 8: Response Surface for Retained Tensile Strength.

The defining criterion of the susceptibility of an asphalt mixture advised by AASHTO T283/89 [2] is of 70% RTS. On AASHTO T283/99 [2], a version made compatible with the SUPERPAVE method, the criterion is off 80% RTS.

The mixtures that obtained the best performance were the ones with 1% of EVA's residue and until 2 hours of aging, obtaining productivity of almost 100%. Only the mixtures with about 2% of EVAR and 3 hours of aging met the 70% AASHTO's RTS criterion.

The other percentages indicate resistance loss of more than 30% when subject to conditioning, highlighting that these mixtures are more susceptible to moisture damage, being able to cause of problems of disaggregation of the aggregates.

4. CONCLUSIONS

One of the main factors that we can emphasize about this research was the fact that EVA's residue was classified as a hazardous waste (Class I). This makes that all the discard's process of this material needs to be reconsidered due to its toxicity and hazardousness when it is discarded in inappropriate ways.

Making use of EVA's residue can be seen as an adequate and interesting alternative both from an economic point of view, since it adds good properties to Petroleum Asphalt Cement, and an environmental point of view, for promoting the reuse of this residue.

Generally speaking, the binder modified with EVA residue showed a satisfactory behavior regarding the physical properties of Petroleum Asphalt Cement and it is within the acceptable range according to the Brazilian norms of paving use. The obtained results are quite promising and should serve as encouragement to the development of other researchers in this area.

Therefore, we can conclude that for the EVA's residues of the footwear industry of Campina Grande, with size-distribution of 2,00mm and incorporated in the conditions of time, temperature and speed of this study, the mixtures that met every requirement were the ones with until 2% of EVA's residue and until 3 hours of aging, achieving a good behavior from the asphalt mixture.

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