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## **ASPHALT BINDER INFLUENCE ON CHEMICAL RESISTANCE OF HMAS: A THEORETICAL AND EXPERIMENTAL STUDY**

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### **ABSTRACT**

As is well known, Hazardous materials (Hazmats) transportation by trucks can represent far more danger than any other truck transportation in terms of accidents.

In particular, when fuel or lubricant, for example, accidentally comes into contact with asphalt pavement for a longer period of time, the asphalt binder is softened, leading to aggregate loss on the surface.

This process can require the replacement of the HMA (Hot Mix Asphalt) course and it depends on many parameters: mix effective porosity, diameter of the pores, quantity and type of fuel, course thickness, immersion time, asphalt binder characteristics, size and shape of the flow paths, etc.

Though important studies on this topic have been proposed, some new and old issues on pavement management still need answers.

In particular, it is not clear how asphalt binder properties can affect these phenomena.

Given this, this paper deals with models and strategies for the analysis of the consequences of Hazmat (in particular, fuel) releases on hot mix asphalts.

A model has been formalized and experimentally validated.

Results demonstrate that, especially for some classes of hazmats, asphalt binder properties and HMA volumetrics are the basis for the interpretation of the involved phenomena and this can be useful in deciding the suitable typology of Hot Mix Asphalt to use in areas when conditions of high vulnerability or/and high probability do occur.

*Keywords: Hazmat, chemical resistance, pavement, porosity, asphalt binder content.*

## 1. BACKGROUND INFORMATION AND MODELING

Though important works on HMA (Hot Mix Asphalt) chemical resistance have been proposed (Maarten et al. 2002, Steenberg et al. 2000, Van Rooijen et al. 2004), some new and old issues still need answers; for this reason, a model has been formalised and validated. When a given mass of fuel is poured into a pavement, the starting mass  $M_0$  (control sample or test specimen) decreases due to the loss of aggregates ( $AG$ ) and asphalt binder ( $B$ ), though small quantities ( $F$ ) of fuel still can remain entrapped in the specimen. The following equation can be considered:

$$M_d = M_0 - B + F - AG \cong M_0 - \Delta M \quad (\text{Eq. 1})$$

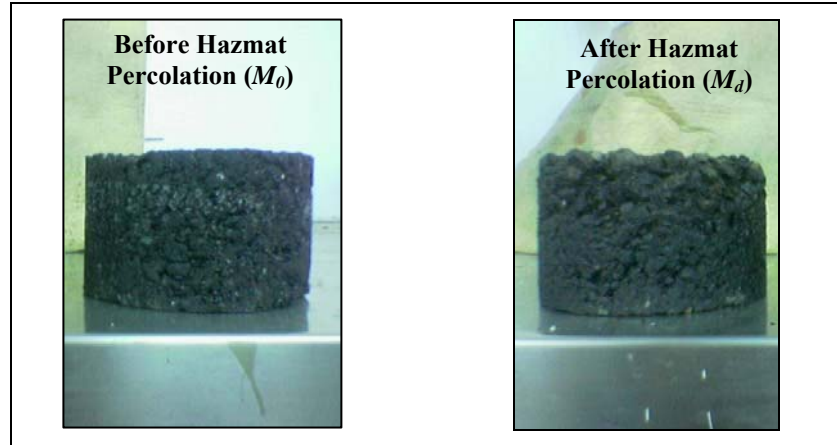
where  $M_d$  is the mass of the dry sample after hazmat percolation or soaking in the fuel.

For the sample showed in Figure 1, the (internal) Volume exposed to the fuel is:

$$V_p = V \cdot n_{eff} = \sum_j \frac{\pi \phi_j^2 l_j}{4} = \sum_j \pi \phi_j l_j \cdot \frac{\phi_j}{4} \quad (\text{Eq. 2})$$

where  $V$  is the total volume of the control sample,  $n_{eff}$  is the effective porosity,  $\phi_j$  and  $l_j$  are the diameter and the length of the  $j$ -th internal pore. Note that according to (Kandhal 1992, Torres 2004), the diameters  $\phi_j$  usually range up to 5 mm c.a, while, for  $\phi_j$  below 0,05 microns, neither asphalt binder flows nor absorption take place. The following expression can be derived (where  $\phi^*$  is a reference diameter of the pores):

$$V_p = \sum_j \frac{\pi \phi_j^2 l_j}{4} = \frac{\phi^*}{4} \sum_j \pi \phi_j l_j \quad (\text{Eq. 3})$$



**Figure 1 Modeling Hazmat percolation in Hot Mix Asphalt.**

On the other hand, the Total Exposed Surface  $S_{Te}$  may be expressed as:

$$S_{Te} \cong S_p + S_e \quad (\text{Eq. 4})$$

where  $S_p$  refers to the (internal) Surface of the exposed Pores and  $S_e$  is the External exposed Surface. By referring to equations (2) and (3) the Surface of the exposed Pores  $S_p$  may be expressed as follows:

$$S_p = \sum_j \pi \phi_j l_j = \frac{4n_{eff}V}{\phi^*} \quad (\text{Eq. 5})$$

If  $\sum_i \phi_i^* \pi$  may be considered negligible (when compared to  $r^2 \cdot \pi$ ), the external exposed Surface is expressed as:

$$S_e = 2\alpha \pi r h + \pi r^2 \quad (\text{Eq. 6})$$

where  $r$  is the radius of the sample and  $\alpha$  is  $h_F$  to  $h$  ratio ( $0 \leq \alpha \leq 1$ );  $h_F$  is the height of the fuel around the sample and  $h$  is sample height. The coefficient  $\alpha$  can be negligible for in-site spillage on dense-graded friction courses. Now, suppose that the Total Exposed Surface,  $S_{Te}$ , can be expressed as:

$$S_{Te} = a \cdot \frac{\Delta V}{\Delta t} \quad (\text{Eq. 7})$$

In this basic equation,  $\Delta t$  is the Exposition Time,  $\Delta V$  is the volume loss caused by hazmat percolation into the sample and the parameter  $a$  (which has the dimensions of the reciprocal of a speed) depends on the particular asphalt binder, size and shape of the flow paths, fuel characteristics, etc. From the equations (4) to (6), the following expression can be obtained:

$$\frac{\Delta V}{V} = \frac{\Delta t}{a} \left( \frac{4n_{eff}}{\phi^*} + \left( \frac{2\alpha}{r} + \frac{1}{h} \right) \right) \quad (\text{Eq. 8})$$

where  $V = \pi \cdot r^2 \cdot h$  is the volume of the sample.

The above formalized model can be used to find a physical-based expression for the parameter  $A$  introduced in the standard EN 12697-43:2005, and there defined as the loss of mass after soaking in fuel:

$$A = \frac{\sum_{i=1}^3 A_i}{3} ; \quad A_i = \frac{m_{1,i} - m_{2,i}}{m_{1,i}} \cdot 100 \quad (\text{Eq. 9; Eq. 10})$$

where  $m_{1,i}$  = initial dry mass of the specimen  $i$  the for soaking in fuel, in grams (g);  $m_{2,i}$  = mass of the dry test specimen  $i$  after soaking in fuel, in grams (g). In the hypothesis of  $F \cong 0$ , if  $i$  refers to the  $i$ -th sample, it is possible to write:

$$\frac{A_i}{100} = \frac{M_{0i} - M_{di}}{M_{0i}} = \frac{\Delta M_i}{M_{0i}} \cong \frac{\Delta V_i \cdot \gamma_L}{V_i \cdot \gamma_{cb}} \quad (\text{Eq. 11})$$

where  $\gamma_L$  = specific gravity of the loss mass (in practice  $\gamma_L$  can range from  $1 \text{ g/cm}^3$  c.a. up to  $3 \text{ g/cm}^3$  c.a);  $\gamma_{cb}$  = specific gravity of the specimen. By referring to equations (8) and (11), the following expression can be derived:

$$\frac{A_i}{100} = \frac{\Delta M_i}{M_{0i}} = \frac{\Delta V_i \cdot \gamma_L}{V_i \cdot \gamma_{cb}} = \frac{\Delta t}{a} \left( \frac{4n_{eff}}{\phi^*} + \left( \frac{2\alpha}{r} + \frac{1}{h} \right) \right) \cdot \frac{\gamma_L}{\gamma_{cb}} \quad (\text{Eq. 12})$$

The equation (12), set forth above, is the basis for the design of experiments below described.

## 2. PROCEDURES AND DEVICES

Experiments have been planned according to the following procedures and standards:

1) Volumetric tests: 1a) %b = asphalt binder content as a percentage of aggregates (B.U. CNR n.38/73; ASTM 6307); carbon tetrachloride has been used as solvent; 1b) G = aggregate gradation (B.U. CNR n. 4/53); 1c) NMA = Nominal Maximum Aggregate Size; 1d) f (%) = filler content ( $d \leq 0.075$  mm); 1e) s(%) = sand content ( $0.075 \text{ mm} \leq d \leq 2$  mm); 1f)  $\gamma_g$  = aggregate apparent specific gravity (B.U. CNR n. 63/78); 1g)  $G_{mb}$  = mix bulk specific gravity (ASTM D6752; ASTM D6857); 1h)  $G_{mbAO}$  = mix bulk specific gravity after opening (ASTM D6752; ASTM D6857); 1i)  $n_{eff}$  = mix effective porosity (ASTM D6752; ASTM D6857);

2) Permeability tests ( $K_{cv}$ ), using a Flexible Wall Permeameter – FWP (ASTM PS 129-01);

3) Brush tests in order to estimate A, B and C (EN 12697-43:2005), where: 3a) A, mean value of the loss of mass after soaking in fuel (Diesel oil), has been defined (together with  $m_{1,i}$  and  $m_{2,i}$ ) in section 1 (see equations (9) and (10)); 3b) B(%) = mean value of the loss of mass after the brush test, where  $B = \sum_i B_i / 3$ , with  $i = 1, 2, 3$  (specimens),  $B_i = ((m_{2,i} - m_{5,i}) / m_{2,i}) \cdot 100$ ,  $m_{5,i}$  = mass of the test specimen  $i$  after soaking and 120 s in the brush test, in grams (g); 3c) C(%) = mean value of the loss of mass of the specimens, where  $C = \sum_i C_i / 3$ , with  $i = 1, 2, 3$  (n° specimen),  $C_i = ((m_{1,i} - m_{5,i}) / m_{1,i}) \cdot 100$ . Note that the parameter C is not defined in the EN standard; it has been introduced in order to have a descriptor able to combine the two different actions (soaking + brushing). Figure 2 shows the main devices used.







Extractors: b(%) (CNR n. 38/73; ASTM 6307)	Pycnometers: $\gamma_g$ (CNR n. 63/78)	Sieves, screens, sieve shaker: $G_s$ (CNR n. 4/53)	Brush tester: A, B, C - (EN 12697- 43:2005)
			
Vacuum Sealing Device: $G_{mb}$ , $G_{mbAO}$ , $n_{eff}$ (ASTM D6752; D6857)	Flexible Wall Permeameter: $k_{cv}$ (ASTM PS 129-01)	$n_{eff} = \frac{G_{mbAO} \cdot \gamma_w - G_{mb} \cdot \gamma_w}{G_{mbAO} \cdot \gamma_w}$ $k_{cv} = R_T \cdot \frac{a_{cs} \cdot l}{A_{cs} \cdot t} \ln \left( \frac{h_1}{h_2} \right)$	
			

Figure 2. Main devices and tests involved (see tables 1 to 3)

Table 1 summarizes the testing procedure used to determine mix specific gravities and porosities. Table 2 summarizes the procedure used to determine the hydraulic conductivity (falling head test). Table 3 and Figures 3 and 4 illustrate the main phases of the test: i) soaking in fuel and removing the fuel; ii) brushing. Note that if A results greater than 5%, then only the first phase is usually performed (poor resistance). When  $A \leq 5\%$ , if  $B < 1\%$  there is good resistance, if  $1 \leq B\% \leq 5\%$  there is moderate resistance, while if  $B > 5\%$  there is still poor resistance to that fuel.

**Table 1 Testing procedure for  $G_{mb}$ ,  $G_{mbAO}$ ,  $n_{eff}$ .**

<ol style="list-style-type: none"> <li>1-Determine the mass of the specimen.</li> <li>2- Open the bag and place the specimen in the vacuum chamber.</li> <li>3- Determine the mass of the sealed specimen in a water bath for determinating <math>G_{mb}</math>.</li> <li>4- Open the bag for allowing the water to penetrate in the specimen to determinate <math>G_{mbAO}</math>.</li> <li>5- Calculate the effective porosity (<math>n_{eff}</math>) from <math>G_{mb}</math> and <math>G_{mbAO}</math>; <math>\gamma_w</math> = water density.</li> </ol>
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**Table 2 Testing procedure of the FWP (Flexible Wall Permeameter).**

<ol style="list-style-type: none"> <li>1 - Loading of the specimen.</li> <li>2 - Apply a confining pressure of <math>96.5 \pm 7.0</math> kPa (<math>14 \pm 1</math> psi).</li> <li>3 - Fill the permeameter with water; an outflow time (<math>\Delta t</math>) is recorded, between two timing marks.</li> </ol>
<p>Note (see figure 2). <math>a_{cs}</math> is the inside cross-sectional area of inlet standpipe (<math>cm^2</math>); <math>l</math> is the thickness of test specimen (cm); <math>A_{cs}</math> is the cross-sectional area of test specimen (<math>cm^2</math>); <math>t</math> is the average elapsed time of water flow between timing marks (s); <math>h_1</math> is the initial hydraulic head on specimen (cm); <math>h_2</math> is the final hydraulic head on specimen (cm); <math>R_T</math> is a coefficient that corrects the calculated k at given temperature to that for <math>20^\circ C</math>.</p>

**Table 3 Brush test (EN 12697-43:2005).**

<p><b>I PHASE: Soaking in fuel; removing the fuel</b></p> <ol style="list-style-type: none"> <li>1 – Record the dry mass of each specimen as <math>m_{1,i}</math>.</li> <li>2 – Place the test specimen in the container; partial immersion of the specimen in the fuel.</li> <li>3 – Cover the container with a metal foil; store the immersed specimen for <math>24 h \pm 30</math> min.</li> <li>4 – Clean the specimen (after the immersion) until the acidity pH of the water is <math>(7,0 \pm 0,5)</math>.</li> <li>5 – Dry the specimen for <math>24 \pm 2</math> h.</li> <li>6- Record the mass (<math>m_{2,i}</math>) (after-drying).</li> <li>7- Calculate A from <math>A_i</math> (%).</li> </ol>
<p><b>II PHASE: <math>A \leq 5\%</math> - Brushing</b></p> <ol style="list-style-type: none"> <li>1- Place the specimen in the steel cylinder of the device.</li> <li>2 - Apply a pressure, <math>P_1</math> or <math>P_2</math>, by a compressor (according to EN 12697-43:2005: <math>P_1</math> bar is a pressure corresponding to the force of <math>(60 \pm 3)N</math> between the brush and the porous asphalt specimen; <math>P_2</math> bar is a pressure corresponding to the force of <math>(140 \pm 5)N</math> between the brush and the non-porous asphalt specimen);</li> <li>3 - Brush test. Total brush time is 120 s: two periods of 30 s and one period of 60 s. After each period the mass of the specimen shall be measured (<math>m_{3,i}</math>, <math>m_{4,i}</math>, <math>m_{5,i}</math>).</li> <li>4- Calculate the mean parameter B from the <math>B_i</math> (%) (<math>i=1, 2, 3</math>).</li> </ol>



Figure 3 Brush test - I PHASE: Soaking in fuel; removing the fuel

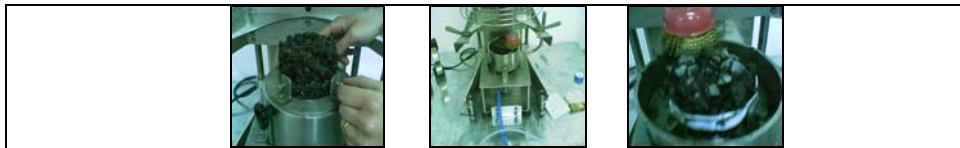


Figure 4 Brush test - II PHASE:  $A \leq 5\%$  - Brushing

### 3. FIRST TASK

During the 1<sup>st</sup> phase all the examined HMAs had the same asphalt binder grade. Owing to the necessity to test the influence of mix parameters over a consistent range of variation, four different bituminous mixes have been tested (see Figure 5, in which mixes acronyms are explained). Average Air voids in DGFCs, BICs and BACs resulted 9%, while, for PEMs, a mean value of 22% has been detected. Each mix type has been subdivided into sets: 7 sets of DGFC (for a total amount of  $7 \times 4 = 28$  specimens), 2 sets of BIC, 2 sets of BAC and 23 sets of PEM. For each set of 4 specimens one of them has been used to control composition parameters and the other three for the Brush test (see Figure 5). Note that, for DGFC, BIC and BAC mixes the prevailing mineral nature of the aggregates was limestone, while for PEMs was basalt.

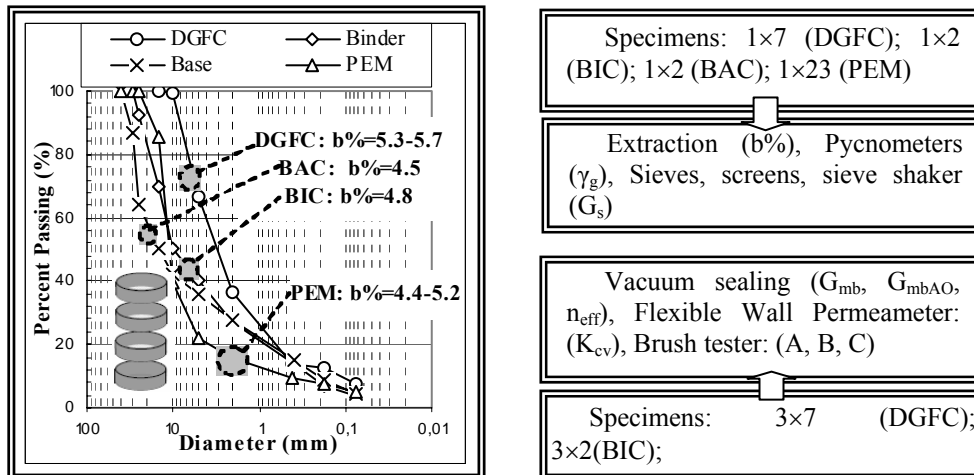


Figure 5-Summary of the design of experiments (BAC = Base Course; BIC= Binder Course; DGFC= Dense Graded Friction Course; PEM = Porous European Mixes).

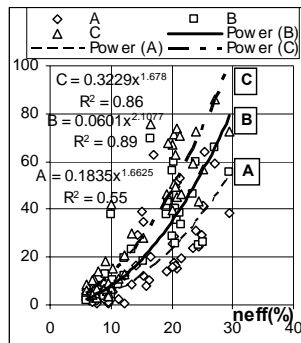
Figures 6 to 11 and table 4 summarize the obtained results.

Figures 6 and 7 deal with the dependence of the effects (selected indicators A, B, C) on composition parameters ( $n_{eff}$ , NMAS); Figure 8 describes the aptitude of hydraulic conductivity to rule such effects, while Figures 9 to 11 summarize the correlation among the selected “effects”.

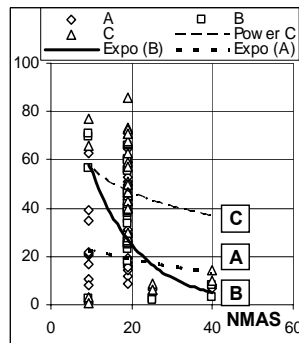
Though the statistic characteristics of the investigation need to be optimized, some observations can be here remarked.

As far as the susceptibility to the effective porosity is concerned (see Figure 6), the obtained results show a strong dependence on  $n_{eff}$  of the mass loss after soaking (A), of the mass loss after brush test (B) and of the mass loss for combined action (soaking + brushing, C). R-square values (logarithmic curves) range from 0.55 (vs A) up to 0.89 (vs B). Linear trendlines give slightly lower R-square values (from 0.37 up to 0.78). When NMAS increases (Figure 7), A, B and C tend to decrease; R-square values are low (up to 0.3) probably due to the insufficient efficiency of NMAS to rule the processes without considering air void content. Note that the significance level of A vs NMAS correlation is relatively large ( $>0.05$ , see table 4); therefore, this correlation may be considered not significant and the two variables are, in practice, not linearly related.

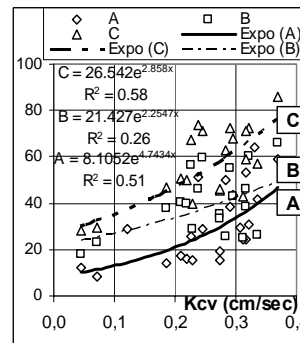
The sensitivity of the three indicators A, B, C to the hydraulic conductivity  $K_{cv}$  (FWP device, PEMs, see Figure 8) seems to be quite satisfactory for A and C ( $R^2=0.51\div0.58$ ), while Brushing susceptibility to  $K_{cv}$  appears low (alike the relative significance). Note that the above-formalised model deals with A (and so, in part, with C), but it doesn't take into account for B. These facts agree with the physical explanation of the involved phenomena: fuel soaking effects depend on percolation efficiency (and so effective porosity, see equation (12)), while brushing effects are mainly an issue of (external) surface tribology and result to be poorly affected by fuel flows in the granular matter.



**Figure 6 - Sensitivity of the parameters A, B, C to the effective porosity  $n_{eff}$ .**



**Figure 7- Sensitivity of the parameters A, B, C to NMAS.**



**Figure 8- Sensitivity of the parameters A, B, C to  $K_{cv}$ .**

Concerning the correlations among the three selected indicators, brushing and soaking susceptibility seem quite well-correlated ( $R^2_{AB}=0.61$ ); of course, the combined susceptibility (Brushing + Soaking) appears to be better related both to the behaviour in

soaking process ( $R^2_{AC}=0.84$ ) and to the descriptor of Brushing process ( $R^2_{BC}=0.93$ ) (see Figures 9 to 11).

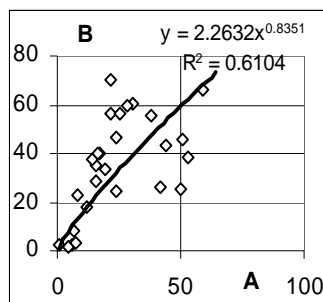


Figure 9- A vs B.

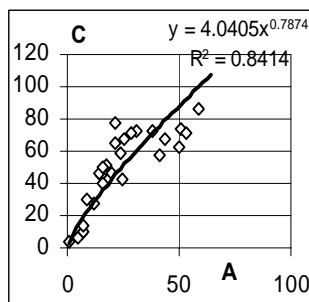


Figure 10- A vs C.

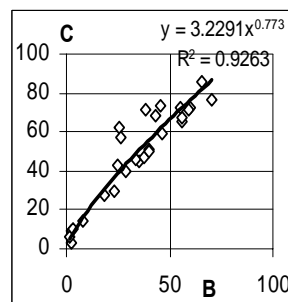


Figure 11- B vs C.

Table 4 Correlations table

Variables		Pearson correlation	Significance (2-tailed)
A	B	0.575	0.020
A	C	0.820	0.000
A	neff	0.609	0.000
A	NMAS	-0.288	0.098
A	Kcv	0.635	0.001
B	C	0.929	0.000
B	neff	0.833	0.000
B	NMAS	-0.520	0.005
B	Kcv	0.484	0.031
C	neff	0.885	0.000
C	NMAS	-0.473	0.013
C	Kcv	0.719	0.000

## 4. 2ND TASK: ASPHALT BINDER INFLUENCE-FIRST EXPERIMENTS

### 4.1 Material characterization

As previously noted, the parameter  $a$  in equation (8) refers mainly to asphalt binder vs fuel (chemical) interaction.

It is important to observe that  $1/a$  is a multiplier of the factors within parenthesis and is susceptible to have an appreciable influence on  $A_i$ .

As a consequence, some attempts have been here performed in order to assess asphalt binder influence on  $A_i$  and to validate the proposed model with reference to the parameter  $a$ .

Therefore, in this second phase, the main aim of the experiments was to assess the influence of the binder characteristics (viscosity in particular) on chemical resistance.



In order to pursue the above-mentioned objectives and scope, asphalt binder properties have been previously analyzed.

The following tests have been performed (see figures 12 to 14):

- a) Penetration test according to CNR BU N.24 -1971 (Norme per l'accettazione dei bitumi per usi Stradali; EN 1426:2007: Bitumen and bituminous binders - Determination of needle penetration); b) Softening point (Ball and ring) according to CNR BU N.35-1973; EN 1427: 2007: Bitumen and bituminous binders - Determination of the softening point - Ring and Ball method); c) Viscosity at 135, 160, 170°C according to ASTM D4402-02 (Standard test method for viscosity determinations of unfilled asphalts using the Brookfield thermosel apparatus; EN 14896:2006: Bitumen and bituminous binders - Dynamic viscosity of bituminous emulsions, cut-back and fluxed bituminous binders -Rotating spindle viscometer method).



**Figure 12- Penetration tester (CNR BU N.24 - 1971)**

**Figure 13- Softening point tester (CNR BU N.35-1973)**

**Figure 14- Brookfield thermosel apparatus (ASTM D4402-02)**

Concerning mat characterization it is important to remark that, on the basis of the obtained results, two main sets of asphalt binder typologies (respectively modified and virgin) seem to be involved (see figures 15 and 16): a) the first group includes asphalt binders with low penetration (pen, 35 dmm c.a), high softening point (PA, 70°C circa), high viscosity ( $\eta$ ,  $\cong 0.3$  Pas at 170°C,  $\cong 0.4$  Pas at 160°C,  $\cong 0.8$  Pas at 135°C); b) the second group includes asphalt binders with high penetration grade ( $\cong 65$  dmm), quite low softening point (45~50°C), quite low viscosity ( $\cong 0.1$  Pas at 170°C,  $\cong 0.15$  Pas at 160°C,  $\cong 0.4$  Pas at 135°C).

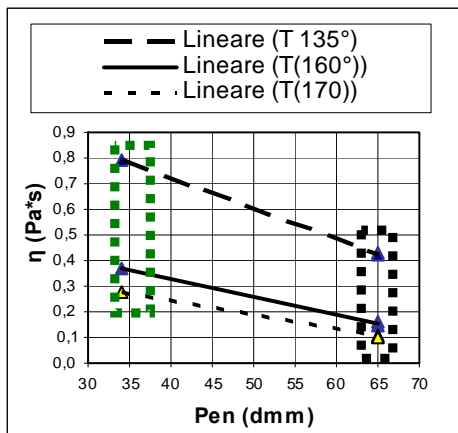


Figure 15 Pen vs  $\eta$

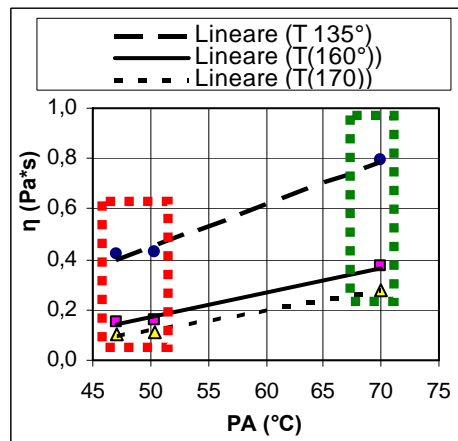


Figure 16 PA vs  $\eta$

Following the model above discussed, in the design of experiments attention has been paid to the control of the other main factors affecting chemical resistance ( $n_{\text{eff}}$ , etc.)

## 4.2 First results

After asphalt binder characterization, the selected specimens have been subjected to the Brush test. Experiments are still in progress.

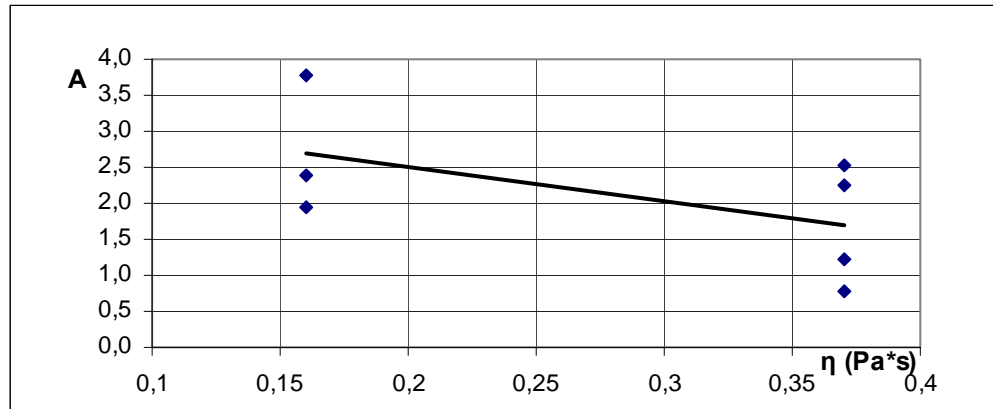
First experiments didn't give reliable results on this topic and more research is still needed.

Figure 17 shows that, for the tested samples, as  $\eta$  increases then A decreases.

It is alike that the hardness of the asphalt binder offered, in this case, more chemical resistance.

Note that:

- a) the number of performed tests is, at this point of the research, still quite insufficient;
- b) an average value of viscosity has been used (see figures 15, 16, 17).

Figure 17- A vs  $\eta$  (Pa\*s)

## 5. FUTURE RESEARCH

First experiments suggested that more research is still needed to better characterize the real influence of the parameter  $a$ , which represents the coefficient of proportionality between the total surface exposed to the action of the fuel (or, more in general, of the hazmat) and the estimate of the relative damage in the unit of time (loss of volume per unit of time).

There are some evidences that the influence of the remaining factors could be too relevant also for the small variations experimentally considered (insufficient control of the boundary conditions).

Experiments are in progress, starting from a different and more basic point of view: analysing the effect of a given quantity/quality of fuel on a given quantity/quality of asphalt binder.

More basic and fundamental descriptors (such as solubility, etc.) are going to be used together with the indicators traditionally used in the field of road materials.

## 6. MAIN FINDINGS

This paper outlines a chemical susceptibility model and preliminary criteria for predictions in the field of HMA chemical resistance; validations have been carried out by a new device called brush tester.

The following conclusions may be drawn:

- i) physical-based model demonstrates that the processes of mass loss after fuel soaking depend on the effective porosity and on asphalt binder vs fuel characteristics;
- ii) the composition parameters of Hot Mix Asphalts (asphalt binder content, filler content, etc...) can affect chemical resistance; anyhow, correlations resulted unsatisfactory;

- iii) hydraulic conductivity has a good correlation with the mass loss after fuel soaking;
- iv) the mass loss after soaking and brushing (C) is well-correlated with the other descriptors (A and B);
- v) first experiments seem to suggest a possible influence of viscosity on chemical resistance; more research is needed to find fundamental or empirical parameters able to rule the model factor  $a$  (or to optimise/change the model itself); the factor  $a$  still remains the least known among the parameters here studied; chemical investigations are in progress and could be helpful in optimizing the level of knowledge on this strategic topic; future research will be aimed at gaining a better understanding of these topics, improving both process modelling and experimental factorial plan.

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