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## LABORATORY TEST BASED ON SPECIMENS OF ASPHALT MIXTURES MADE OF CRUSHED STEELWORKS SLAG AGGREGATE

BY

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**Abstract.** In recent years, environmental quality studies have shown that the steel industry is one of the industries whose work involves a significant consumption of natural resources and energy, is also generating waste which, because of the potential for recovery can be converted into by-products. This paper will present some experimental data on the development of asphalt mixtures with slag aggregate plants. For these two recipes of asphalt mixtures were made (BAR16, BAD20), for which determinations were made of the laboratory and were set up physical-mechanical properties, main of these mixtures. The results of this review have highlighted the real possibility of using these aggregates as building materials used in asphalt for roads and streets.

**Keywords:** density; absorption; Marshall stability; exfoliation; steelworks.

### 1. Introduction

The problem relation between man and nature is not new. Science and modern technology, immensurable increasing man’s power, have improved the standard of living everywhere. The reverse of industrial civilization, of technical

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and scientific revolution, of material progress was and is the degradation of the natural environment.

Industrial society, on the whole, is harmful to the health of natural systems on which ultimately depends life and doesn't accept the fact that there are limits of the planet to provide resources and absorb industrial waste. Between the growth of the economy and the volume of waste is a direct link which contributes to deepening the conflict between technosphere and ecosphere.

The massive growth of waste of all kinds endangers the quality of environmental factors and the last decades industrial activity was accompanied by pollution phenomena due largely the lack of long term prevision and administration, but especially because the realisation of industrial production has as a consequence uncontrolled loss of materials in various states of aggregation.

To support this idea, laboratory tests were made inside of Faculty of Constructions and Installations from Iasi, that analysed the achievement of asphalt pavement which have in composition artificial aggregates obtained by crushing steelwork slag wastes.

## 2. Laboratory Tests

Steelwork slag aggregates are used mainly in the foundation layers or base layers of the road structures, but can be used also in the asphaltic layers, replacing crushed quarry aggregates.

In order to determine the physic-mechanical characteristics of the asphaltic mixtures there were made a number of 2 asphaltic recipes which have in composition steelwork slug aggregates namely: BAR 16, BAD 20 (Iriciuc, 2011).

The aggregates which are used for the preparation of asphaltic mixtures are natural and artificial aggregates according SR EN 13043-2003 – “Aggregates for bituminous mixtures and surface treatments for roads, airfields and other trafficked areas”.

To achieve these asphalt mixtures recipes were needed the following sorts of aggregates:

- a) crushed slag aggregate 0,...,4 mm;
- b) crushed slag aggregate 4,...,8 mm;
- c) crushed slag aggregate 8,...,16 mm;
- d) crushed slag aggregate 16,...,25 mm;
- e) natural sand 0,...,4 mm;
- f) limestone filler .

Thus, considering the granularity of each aggregate the dosages of the two recipes are presented in Table 1.

**Table 1**  
*The Percentages of Aggregates used to Produce Asphalt Mixes Recipes*

Aggregate / Recipe	BAR16	BAD 20
Limestone filler, [%]	10.00	9.00
Natural sand 0,...,4 mm, [%]	–	10.00
Crushed slag aggregate 0,...,4 mm, [%]	30.00	28.00
Crushed slag aggregate 4,...,8 mm, [%]	25.00	20.00
Crushed slag aggregate 8,...,16 mm, [%]	35.00	15.00
Crushed slag aggregate 16,...,20 mm, [%]	–	18.00

The asphalt mixtures were prepared with a D50/70 bitumen, SR EN12591-2009 – “Bitumen and bituminous binders – Specifications for paving grade bitumen’s” - whose main characteristics are presented in Table 2 (SR EN 12591-2009).

**Table 2**  
*Bitumen characteristics used in the preparation of asphalt recipes*

Bitumen characteristics D 50/70	Values obtained in the laboratory:	SR EN 12591-2009
Penetration at +25oC, 1/10 mm	66	66...70
Softening point (I.B.), [°C]	51.8	49...55
Penetration index I.P.	–0.158	–1,5...+0,7

The penetration was determined according SR EN 1426-2007 – “Bitumen and bituminous binders - Determination of needle penetration”.

To establish the optimal dosage of binder were made Marshall type specimens with different dosages of bitumen, for which were determined the following characteristics:

- a) determination of asphalt mixture density;
- b) determination of water absorption;
- c) determination of Marshall stability.

### 3. Determination of Asphalt Mixture Density

The determination is performed according to SR EN 12697-6 /2012 – “Bituminous mixtures – Test methods for hot mix asphalt – Part 6: Determination of bulk density of bituminous specimens”.

This test is used to determine the apparent mass volume of bituminous specimen after compaction. The method is applicable to specimens compacted in the laboratory.

The results of laboratory measurements are specified in Table 3.

**Table 3***Density Values on the Marshall Specimens*

Bitumen percentage, [%]	Bitumen type	$\rho_a$ , [kg/m <sup>3</sup> ]
BAR16		
5.50	D 50/70	2,690
5.75		2,717
6.00		2,704
BAD20		
5.00	D 50/70	2,644
5.25		2,646
5.50		2,649
5.75		2,671
6.00		2,683
6.25		2,680

#### 4. Determination of Water Absorption

The determination is performed on Marshall cylinders shape specimens made in laboratory respecting SR EN 1097-6:2013 – “Tests for mechanical and physical properties of aggregates – Part 6: Determination of particle density and water absorption”.

Water absorption is the amount of water absorbed by a specimen submerged in water for three hours at a vacuum of 15,....,20 mmHg, and then for 2 hours in the same water at atmospheric pressure. Volume absorption values are shown in Table 4.

**Table 4***Values of Volume Absorption for Marshall Specimens*

Bitumen percentage, [%]	Bitumen type	SR EN 1097-6, [%]	Volume absorption, [%]
BAR16			
5.50	D 50/70	Max. 6.00	3,757
5.75			2,403
6.00			1,995
BAD20			
5.00	D 50/70	Max. 6.00	4,630
5.25			4,233
5.50			2,138
5.75			0,952
6.00			0,724
6.25			0,632

### 5. Determination of Marshall Stability

The determination is performed according to SR EN 12697-34+A1 – “Asphalt mixtures – Test methods for hot asphalt mixtures Marshall testing.”.

Specimens remain first 30,...,35 min. in a water bath at a temperature of  $60 \pm 1^\circ\text{C}$ . Specimen is loaded immediately with a speed of  $50 \pm 3$  mm per minute. When the task indicated by the dial begins to decrease the trying is stop. Maximum load indicated by the dial, expressed in daN, represents Marshall stability and deformation read on the micro comparator, expressed in 1/10 mm, is the value of the flow.

Marshall stability values S, creep I, and the S/I ratio, on the series of asphalt mixtures prepared in the laboratory are presented in Table 5.

**Table 5**  
*Marshall Stability Values S, creep I, and the Ratio S / I*

Bitumen percentage %	Bitumen type	S kN	S, [kN] SR EN 12697-34	I mm	I mm SR EN 12697-34	S/I ratio, kN/mm	S/I ratio, kN/mm SR EN 12697-34
<b>BAR16</b>							
5.50	D 50/70	12.0	6.5,...,13	3.95	1.5...4.0	3.04	Min. 1.6
5.75		15.2		4.25		3.58	
6.00		12.3		4.44		2,770	
<b>BAD20</b>							
5.00	D 50/70	16.0	5.0,...,13	3.44	1.5...4.0	2.48	Min. 2.1
5.25		15.1		3.84		3.93	
5.50		11.6		4.42		2.62	
5.75		16.4		5.08		3.22	
6.00		13.5		5.22		2.59	
6.25		11.5		5.59		2.05	

The optimum dosage of bitumen determined from laboratory tests and on Marshall samples are close to the ones in the standard (Table 6).

**Table 6**  
*Values of bitumen dosages after laboratory tests results*

Type of asphalt mixture	Bitumen dosages after laboratory tests results, [%]	Recommended bitumen dosage values
BAR16	5.75	5.7...6.2 %
BAD20	5.50	5.5...7.0 %

After establishing the optimal dosage of bitumen, asphalt specimens were made for determination the freeze-thaw phenomenon and Wheel tracking.

## 6. Asphaltic Specimens Testing at Freeze-Thaw Phenomenon

To determine the physical - mechanical properties of asphalt mixtures at the freeze-thaw phenomenon, recipes of asphalt mixtures were prepared in the laboratory with optimal bitumen dosage resulted in the laboratory. Test specimens resulting from asphalt mixtures: BAR 16 and BAD20.

Some of the samples were used to determine the freeze-thaw phenomenon at 28 days, while the other samples were witness samples that were tested in comparison at swelling, Marshall stability, creep index and the S/I ratio.

The first test samples were subjected to 28 freeze-thaw cycles in Atica TZ1 solution. This solution is a chemical fondant containing NaCl and molasses, solution prepared by the company "ATICA CHEMICALS SRL Râmnicu Vâlcea." The samples used were held during the freeze-thaw cycles for 10 hours at the positive (+18,...,+22°C) and 14 hours at negative temperatures (-21°C) (Anton, 2008; SR EN 12697-41:2006).

The results obtained at swelling, Marshall stability, flow index, S/I ratio, for the samples used in freeze-thaw action are presented in Table 7.

**Table 7**  
*Atica TZ1 solution treated samples to 28 days*

Mixture	Atica TZ1 solution treated samples to 28 days			
	Swelling, %	Stability (S) at 60°C, kN	Flow index (I), mm	S/I ratio, kN/mm
BAR16	0.195	9,90	4,40	2,25
BAD20	0.285	10,9	4,29	2,54

Comparative measurements were made on control samples to determine swelling to 28 days, Marshall stability, flow index, S/I ratio. The results are presented in Table 8.

Analyzing the two tables, it can be concluded that aggregates of crushed slag from steelworks used to produce asphalt mixtures, did not suffered significantly changes after 28 freeze-thaw cycles. That means the values of the swelling, Marshall stability and creep is substantially equal and after freeze-thaw action.

**Table 8**  
*Control Samples at 28 Days (normal conditions)*

Mixture	Control samples at 28 days			
	Swelling, [%]	Stability (S) at 60°C, kN	Flow index (I) mm	S/I ratio kN/mm
BAR16	0.121	9.80	4.41	2.22
BAD20	0.270	12.1	4.73	2.55

Determination of the affinity between crushed slag from steelworks and bitumen (SR 12697-11:2003).

This determination is made under the standard SR 12697-11:2003 – Asphaltic mixtures – Testing methods for poured hot asphaltic mixtures. Part 11 – The determination of the affinity between the aggregate and bitumen.

This European standard establishes procedures for the determination of the affinity between the aggregate and bitumen, as well its influence on the combination peel susceptibility.

The combination peel susceptibility, as determined by this procedures, is an indirect action of the adhesiveness of a binder to various aggregates or various binders adhere to certain aggregates. The procedure can be also used for evaluating the effect of humidity upon a given aggregate-binder combination, with or without additives including liquids, for example amines or fillers (ex: hydrated chalk or cement).

The affinity between aggregates and bitumen represents the bitumen coverage of loose aggregates particles topping with bitumen determined visually after the effect of mechanical stirring in the presence of water.

The percentage of exfoliation represents the average proportion of the aggregates particles surface from which the binder was washed due to water, expressed in percentages.

The aggregate is sifted in accordance with the provisions of EN 12679-2. The part between 7 mm up to 14 mm (or alternatively another part) is washed, dried and mixed with bitumen to obtain a total uniform topping. The coating aggregates are subject to delamination in boiled water in specified conditions, using a simple device in which local overheating may not occur. By contact with a chemical reagent, whose consumption is proportional to the aggregates intopping surface, the coating degree with bitumen is determined with reference to a calibration curve established by a well-defined procedure.

For limestone aggregates the reagent used is spirit of salt and for silico-limestone and siliceous aggregates the reagent used is hydrogen fluoride.

For the silico-limestone and siliceous aggregates proceed as follows:

a) put each of the mixtures of 200 g in contact for (1 h ± 1 min) with (200 ± 0.1 g) of hydrogen fluoride in a 800 ml polyethylene cup covered with a

cover glass made of glass protected by a thin film of plastic or wax. The acid attack is carried out with shaking using a vibrating shaker. The solution is homogenized at the beginning and after an hour through several rotations of the cup;

b) after the contact time has expired, homogenize and separate the hydrogen fluoride from the aggregate by gently pouring the solution in a 250 ml polyethylene graduated cylinder. Titrate with potassium hydroxide in the presence of the phenolphthalein on a aliquot side of 25 ml. Repeat titration on a second aliquot side of 25 ml. Determine the medium volume in ml of potassium hydroxide necessary for titration by rounding to the nearest value of 0.05 ml;

c) settle 200 ( $\pm 0.5$ ) g of the coating aggregate on a metal surface (for example a metallic box cover) and weight this material with a rounding to the nearest value of 0.1 g resulting mass ( $M_1$ );

d) immediately the water starts boiling, pour the 200 g of coating aggregate on the net support in the cup having the hands protected with rubber gloves and start the chronometer;

e) bring the water quickly again at boiling (1 min, maximum 2 min) and leave it to boil for 10 minutes;

f) remove the bitumen that maintains at the surface by introducing in the cup cold water below the surface of the cold water, pour the water and empty the cup on a 7mm sieve. Leave some time for the aggregate to dry and cool down;

g) transfer the aggregate in a 800 ml dry cup (of glass or polyethylene, based on used acid) and weight by rounding to the nearest division of 0.1 g resulting mass ( $M_2$ );

h) determine the medium volume in ml necessary for neutralization by rounding to the nearest value of 0.05 ml.

Calculating the coating degree.

For the exfoliating test, calculate the volume of consumed acid ( $A_c$ ) in ml, rounded to the nearest value of 0.05 ml, from the following formula:

$$A_c = 25.0 - \frac{M_2 - M_1}{200} r V, \quad (1)$$

where:  $M_1$  is the coating aggregate mass (in grams), rounded to the nearest value of 0.1g;  $M_2$  – wet coating aggregate mass after test (in grams), rounded to the nearest value of 0.1g;  $r$  – acid/base equivalency factor;  $V$  – medium volume of 0.1 N NaOH or N KOH necessary to neutralize the 25 ml acid portion at the exfoliation test (in ml) rounded to the nearest value of 0.05 ml.

i) represent the  $A_c$  value on the calibration curve and read the corresponding exfoliated aggregates percentage. This value, rounded to the



nearest unitary value, represents the exfoliation percentage (SR EN 12697-11:2006);

j) calculate the average of the two exfoliation percentages obtained from the tests, rounded to the nearest unitary value. If the individual values differ from this average value by more than 5% in absolute terms, there will be a third exfoliation test and recalculate the average value for the two corresponding tests;

k) calculate the coating degree as  $100 - \text{exfoliation } \%$ .

For crushed steelworks slag (DSU Galati source), the value  $A_c = 8.2$  for the first test and respectively 7.9 for the second test, where results from the calibration curve an average exfoliation percentage of 7%.

Use of steelworks slag aggregates in the road technology leads to numerous technical and economic advantages for both the user and the manufacturer:

- i) diversification of road construction materials;
- ii) reducing construction costs by replacing natural aggregates;
- iii) elimination of waste disposal dumps made from steel mills and therefore environmental protection;
- iv) properties of steelworks slag aggregates allow the road construction without affecting the environment at weather factors (rain, freezing-thawing.);
- v) ensuring the service life of roads safe and comfortable, due to texture that provides roughness and high resistance to skidding.

In terms of laboratory testing can be concluded the following:

1. percentages of steelworks slag aggregate used in asphalt mixtures making follow the recommendations of standard;
2. optimum binder content was determined taking into account asphalt mixture density, water absorption, stability of the Marshall specimens; resulted dosage in the preparation of bituminous asphalt mixtures with steelworks slag aggregate is generally lower than the recommended dosage for a classical mixture;
3. bulk density of the asphalt mixture varies between 2.6 and 2.7  $\text{kg/m}^3$ , being higher than the bulk density of a classical mixture. The difference is made by the higher density of steelworks slag aggregates compared to the density of natural aggregates;
4. in the laboratory tests for determining Marshall stability can be observed that the values of stability S, creep I and the S / I ratio is less than the maximum imposed by the standard;
5. after testing the freeze-thaw phenomenon, after the 28 freeze-thaw cycles, it was found that the differences between a witness mixture with steelworks slag aggregates and a mixture with steelworks slag aggregates

treated with a solution of Attica TZ1, does not present significant differences in terms of swelling and Marshall stability;

6. after performing wheel tracking test, we can observe that the asphaltic layers made with steelwork slug crushed aggregates have depths of the paths under the maximum values allowed by the standard.

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## ÎNCERCĂRI DE LABORATOR PE EPRUVETE DIN MIXTURI ASFALTICE REALIZATE CU AGREGATE DIN ZGURĂ CONCASATĂ DE OȚELĂRIE

(Rezumat)

În ultimii ani, studiile privind calitatea mediului au arătat că industria siderurgică este una dintre industriile a căror muncă implică un consum semnificativ de resurse naturale și energie, este, de asemenea, generatoare de deșeuri care, din cauza potențialului de recuperare pot fi transformate în produse secundare. Această lucrare prezintă unele date experimentale asupra preparării mixturii asfaltice cu zgură. Pentru aceste doua rețete de mixtură asfaltică s-au făcut două amestecuri (BAR16, BAD20).

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Determinările au fost făcute în laborator și au fost stabilite proprietățile fizico-mecanice. Rezultatele acestor analize au subliniat posibilitatea reală de a folosi aceste agregate din zgură în mixturile asfaltice pentru drumuri și străzi.

