Characteristics of bituminous shales used in asphalt mixtures

D-Dr Hanganu

1) Gheorghe Asachi Technical University of Iasi, Faculty of Civil Engineering and Building Services, Bd. Dimitrie Mangeron, no 43, 700050, city of Iasi, Romania

E-mail: hanganu.dragos@gmail.com

Abstract. Bituminous shales, bitumolites or pyrosists are sedimentary rocks impregnated with an insolvent bitumen. In Romania, studies have been carried out on bituminous shales by almost all Romanian geologists, starting with Cobâlcescu in 1877, continuing with Mrazec in 1907, Athanasieiu in 1926, Popescu-Voitești in 1943, etc. In 1956, Nicolae Grigoraș was the one who made the first studies in detail on bituminous shales. Considering that the only norm on the basis of which the technological recipes for asphalt mixtures with bituminous sand are made, it underwent the last modifications in 1985, one of the specific objectives of the research was to update the technical specifications contained in the regulations in order to streamline the technological process associated with these mixtures and for a better implementation at national level of these practices. Without this long-awaited review, the use of this unconventional material would become more difficult, although it has both technical, economic and environmental benefits.

1. Introduction
Several studies were carried out in Romania by scientists regarding bituminous shales [1, 4, 8]. Considering that the only regulation which details the technological recipes for asphalt mixtures with bituminous sand are made, underwent numerous modifications, the last one occurred in 1985 [7]. Without this long-awaited review, the use of this unconventional material would become more difficult, although it has both technical, economic and environmental benefits [2, 6].
The bituminous shales were previously ground, after which the crushing sand was used to prepare the types of asphalt mixture type AC 22.4 BIN 50/70 SI AC 16 SURF 50/70. After the preparation of the asphalt mixtures, the physical-mechanical characteristics were determined.

Through her study, Ionita G, experimental and theoretical activities were carried out, undertaken, in order to acquire new knowledge on the specified materials, respectively the crushing sand used to prepare asphalt mixtures and based on observations, tests and interpretation of laboratory results, which, based on some knowledge already existing, will lead to a significant improvement of the knowledge of applicability of these materials in a specific field, respectively that of construction materials [9].

This research is not carried out for the purpose of direct commercial use but for the purpose of enriching the consciousness in the scientific sphere in terms of a topical material, but poorly capitalized in road construction.

2. Design
In order to design an asphalt mixture recipe, respectively to establish the best proportions of the component materials so as to obtain its best characteristics, it is necessary to analyse the component materials considering that the properties of both mineral aggregates and binder influences differently the properties of the mixture. The components of the asphalt mixture will be analysed first, in order to take into account all the premises for the elaboration of the study [5].

2.1. Natural mineral aggregates
The mineral aggregates used to obtain the asphalt mixtures come from stone quarries or gravel pits. Their destination may differ depending on the destination of the respective asphalt mixture. In general,
the quarry aggregates are used on technical class roads I-III (high-traffic roads such as highways, express roads, national roads) and the gravel roads are used in the construction of lower technical class roads, IV-V (county roads, communal, local roads) [3].

In the present study, mineral aggregates from the Suseni quarry - Harghita county and natural sand from the Tupilați gravel pit - Neamț county will be used.

2.2. Bitumen
In order to obtain an asphalt concrete, this being a mixture of agglomerated mineral materials with a bituminous binder, plain petroleum bitumen for roads, non-paraffin, class 50/70 was used.

2.3. The filler
The granularity of the filler required to determine the weight in the dosing study is shown in table 1.

Table 1. The granularity of the limestone filler

| Nr. crt. | Characteristic | Limestone filler |
|----------|----------------|------------------|
| 1        | Granularity (passes % through sieve of ... mm) | Average test |
| 2        | 0,125          | 100              |
|          | 0,063          | 87,5             |
|          | Average        | 75,4             |

2.4. The share of mineral aggregates in asphalt mixtures.
The aim is to obtain high-performance asphalt mixtures, in which the mineral skeleton consists of sorts of natural mineral aggregate quarry (Suseni quarry) and crushing sand from bituminous shale, useful for making asphalt layers of wear and bonding.

AC 16 SURF 50/70 and AC 22.4 BIN 50/70, of asphalt concrete type with chipping - hereinafter referred to as asphalt mixtures with bituminous crush sand for wear and connection layers of road systems.

The preparation of the asphalt mixtures was performed in the laboratory, by manually mixing all the components, Figure 1, at the temperatures specified in table 2 until homogeneous mixtures were obtained, completely coated, which were then processed.

Table 2. Temperatures of components and asphalt mixture in preparation

| Nr. crt. | Bitumen type | Bitumen temperature, °C | Mineral aggregate temperature, °C | Asphalt concrete temperature at preparation, °C |
|----------|--------------|-------------------------|----------------------------------|-----------------------------------------------|
| 1        | D 50/70      | 150 - 170               | 140 - 190                        | 140 - 180                                     |

Individually constituted batches were made for each proposed dosage, a load that initially had a set weight of 9500 g, the weight required to obtain 8 Marshall cylinders from each type of asphalt mixture, cylinders that were subsequently tested.
2.5. Bitumen dosage

Asphalt mixture samples, Figure 2, consisting of asphalt mixture are:

I. AC 16 SURF 50/70 with bituminous shale crushing sand for wear layers, series: 300-307; 310-317; 320-327, at which the bitumen dosage differs: 5.7%, 5.9% respectively 6.1%.

II. AC 22.4 BIN 50/70 with bituminous shale crushing sand for connecting layers, series: 600-607; 610-617; 620-627, at which the bitumen dosage differs: 4.3%, 4.5% and 4.7% respectively

Marshall cylinders were made from the asphalt mixture thus prepared, applying 50 ram blows on each face. The specimens were stored in laboratory conditions, at room temperature, until completely cooled, Figure 3.

Figure 1. Mixing aggregates and manual bitumen in the laboratory

Figure 2. Marshall cylinders made of AC 16 SURF 50/70 asphalt mixture with bituminous shale crushing sand from Village Nechit, Neamt county
**Figure 3.** Marshall cylinders made of AC 22.4 BIN 50/70 asphalt mixture with crushed sand from bituminous shales from Nechit Village, Neamt County

### 3. Tests performed:

#### 3.1. Bulk density

The ratio between the mass of the unit volume of the compacted asphalt mixture specimen, including the gaps filled with air, and the volume of the specimen, expressed in g / cm³, determined on specimens kept for one hour in water at 20 °C.

Tables 3 and 4 show the values obtained of the apparent densities on the series of cylinders from the asphalt mixture with crushing sand from bituminous shale. The variation of the apparent densities of the three dosage variants is shown in the diagrams in figures 4 and 5.

**Table 3.** Results for bulk density on AC 16 SURF 50/70 bituminous sand crushing mixture

| Asphalt mixture type                                      | Bitumen dosage, % | Cylinder series | Apparent density, g / cm³ | AC 16 SURF 50/70 Standard |
|----------------------------------------------------------|-------------------|-----------------|----------------------------|---------------------------|
| AC 16 SURF 50/70 with bituminous shale crushing sand     | 5,7               | 300-307         | 2,351                      | 2,376                      |
| AC 16 SURF 50/70 with bituminous shale crushing sand     | 5,9               | 310-317         | 2,370                      | 2,376                      |
| AC 16 SURF 50/70 with bituminous shale crushing sand     | 6,1               | 320-327         | 2,376                      | 2,376                      |
Figure 4. Diagram of variation of apparent densities for asphalt mixture with crushing sand from bituminous shale type AC 16 SURF 50/70

Table 4. Results obtained for the apparent density on the bituminous shale sand crushing mixture type AC 22.4 BIN 50/70

| Asphalt mixture type | Bitumen dosage, % | Cylinder series | Apparent density, g/cm³ | AC 22.4 BIN 50/70 Standard |
|----------------------|-------------------|----------------|-------------------------|--------------------------|
| AC 22.4 BIN 50/70 with bituminous shale crushing sand | 4,3 | 600-607 | 2,339 | 2,368 |
| AC 22.4 BIN 50/70 with bituminous shale crushing sand | 4,5 | 610-617 | 2,365 | 2,368 |
| AC 22.4 BIN 50/70 with bituminous shale crushing sand | 4,7 | 620-627 | 2,399 | 2,368 |
3.2. Water absorption

The amount of water absorbed by the specimens is measured after the sample is immersed in water for 3 hours at a vacuum of 15 ... 20 mmHg, followed by a time of 2 hours maintained in water at atmospheric pressure, expressed in % vol.

Tables 5 and 6 give the obtained values of the water absorption on the series of cylinders from the asphalt mixture with crushing sand from bituminous shale. The variation of the apparent densities of the three dosage variants is shown in the diagrams from figures 6 and 7.

**Table 5.** Results obtained for water absorption on asphalt mixture with crushed sand from bituminous shale AC 16 SURF 50/70

| Asphalt mixture type                      | Bitumen dosage, % | Cylinder series | Water absorption, g / cm³ | AC 16 SURF 50/70 Standard |
|------------------------------------------|-------------------|-----------------|---------------------------|--------------------------|
| AC 16 SURF 50/70 with bituminous shale crushing sand | 5,7               | 300-307         | 1,065                     | 1,80                     |
| AC 16 SURF 50/70 with bituminous shale crushing sand | 5,9               | 310-317         | 0,686                     | 1,80                     |
| AC 16 SURF 50/70 with bituminous shale crushing sand | 6,1               | 320-327         | 0,202                     | 1,80                     |
Figure 6. Diagram of variation of water absorption for asphalt mixture with crushing sand from bituminous shale BA16

Table 6. Results obtained for water absorption on asphalt mixture with crushing sand from bituminous shale AC 22.4 BIN 50/70

| Asphalt mixture type                                      | Bitumen dosage,% | Cylinder series | Water absorption, g / cm³ | AC 22.4 BIN 50/70 Standard |
|-----------------------------------------------------------|------------------|-----------------|----------------------------|---------------------------|
| AC 22.4 BIN 50/70 with bituminous shale crushing sand     | 4,3              | 600-607         | 2,900                      | 2,90                       |
| AC 22.4 BIN 50/70 with bituminous shale crushing sand     | 4,5              | 610-617         | 2,154                      | 2,90                       |
| AC 22.4 BIN 50/70 with bituminous shale crushing sand     | 4,7              | 620-627         | 1,915                      | 2,90                       |
3.3. Marshall stability

Is assessed by the force reached when the test specimen breaks, expressed in KN.

Tables 7 and 8 give the obtained values of the Marshall stability on the series of cylinders from the asphalt mixture with crushing sand from bituminous shale. The variation of the Marshall stability of the three dosage samples is shown in the diagrams presented in figures 8 and 9.

**Table 7.** Results for Marshall stability on AC 16 SURF 50/70 asphalt mixture with bituminous shale crushing sand

| Asphalt mixture type                      | Bitumen dosage, % | Cylinder series | Stability Marshall, KN | AC 16 SURF 50/70 Standard |
|-------------------------------------------|-------------------|----------------|------------------------|----------------------------|
| AC 16 SURF 50/70 with bituminous shale crushing sand | 5,7               | 300-307        | 8,8                    | 10,30                      |
| AC 16 SURF 50/70 with bituminous shale crushing sand | 5,9               | 310-317        | 8,0                    | 10,30                      |
| AC 16 SURF 50/70 with bituminous shale crushing sand | 6,1               | 320-327        | 7,4                    | 10,30                      |
Figure 8. Marshall stability variation diagram on asphalt mixture AC 16 SURF 50/70 with bituminous shale crushing sand.

Table 8. Results obtained for Marshall stability on AC 22.4 BIN 50/70 asphalt mixture with bituminous shale crushing sand

| Asphalt mixture type                      | Bitumen dosage,% | Cylinder series | Stability Marshall, KN | AC 22.4 BIN 50/70 Standard |
|------------------------------------------|------------------|----------------|------------------------|-----------------------------|
| AC 22.4 BIN 50/70 with bituminous shale crushing sand | 4,3 | 600-607 | 8,0 | 8,30 |
| AC 22.4 BIN 50/70 with bituminous shale crushing sand | 4,5 | 610-617 | 8,4 | 8,30 |
| AC 22.4 BIN 50/70 with bituminous shale crushing sand | 4,7 | 620-627 | 9,2 | 8,30 |
3.4. Marshall flow
The Marshall flow refers to the deformation reached when the test specimen breaks and is expressed in mm.

Tables 9 and 10 give the obtained values of Marshall stability on the series of cylinders from the asphalt mixture with crushing sand from bituminous shale. The variation of the Marshall stability of the three dosage variants is shown in the diagrams from figures 10 and 11.

Table 9. Results for Marshall flow on asphalt sand mix with bituminous shale type AC 16 SURF 50/70

| Asphalt mixture type                                      | Bitumen dosage, % | Cylinder series | Flowing, mm | AC 16 SURF 50/70 Standard |
|----------------------------------------------------------|-------------------|----------------|-------------|--------------------------|
| AC 16 SURF 50/70 with bituminous shale crushing sand     | 5,7               | 300-307        | 3,77        | 3,70                     |
| AC 16 SURF 50/70 with bituminous shale crushing sand     | 5,9               | 310-317        | 4,46        | 3,70                     |
| AC 16 SURF 50/70 with bituminous shale crushing sand     | 6,1               | 320-327        | 4,68        | 3,70                     |

Figure 9. Marshall stability variation diagram for asphalt mixture AC 22.4 BIN 50/70 with bituminous shale crushing sand
Figure 10. Marshall flow variation diagram for asphalt mixture with crushing sand from bituminous shale type AC 16 SURF 50/70

Table 10. Results obtained for Marshall flow AC 22.4 BIN 50/70 asphalt mixture with bituminous shale crushing sand

| Asphalt mixture type                                      | Bitumen dosage,% | Cylinder series | Flowing, mm | AC 22.4 BIN 50/70 Standard |
|-----------------------------------------------------------|------------------|----------------|-------------|----------------------------|
| AC 22.4 BIN 50/70 with bituminous shale crushing sand      | 4,3              | 600-607        | 4,15        | 3,30                       |
| AC 22.4 BIN 50/70 with bituminous shale crushing sand      | 4,5              | 610-617        | 4,27        | 3,30                       |
| AC 22.4 BIN 50/70 with bituminous shale crushing sand      | 4,7              | 620-627        | 4,41        | 3,30                       |
Figure 11. Marshall flow variation diagram for AC 22.4 BIN 50/70 mixture with bituminous shale crushing sand

3.5. Swelling in water
The swelling in water phenomenon establishes the harmful influence of constituent components of mineral aggregates that are sensitive to swelling - clay, by keeping them in water for 28 days [6]. Swelling was also determined at regular intermediate intervals of 7, 14, 21 and including 28 days. The results obtained at regular intermediate intervals of 7, 14, 21 and even at 28 days showed that the swelling in the water is 0.

4. Conclusions
This report shows solutions that could be put into practice regarding the preparation of asphalt mixtures using bituminous shale crushing sand from Romania. These solutions can be adopted in order to achieve significant savings in bitumen.

For all technological recipes of asphalt mixtures prepared in the laboratory of roads with bituminous shale crushing sand, the values of physical-mechanical characteristics were determined by static tests: bulk density, water absorption, stability at 60 °C, flow index, swelling at 28 days.

The results obtained from the studies performed in the road laboratory are favourable for asphalt mixtures prepared with bituminous shale crushing sand from Nechit Village, Neamt County.

Based on the results obtained from the tests made on the asphalt mixtures with crushing sand from bituminous shales from Village Nechit, it is recommended to use them in the composition of asphalt mixtures, in order to significantly reduce the costs related to the production of asphalt mixtures. This solution is very advantageous especially for areas near natural deposits.

Acknowledgments
Regards to professor Gugionic Gheorghe and professor Crina Miclăuș, for offering technical documentation, support and making this study possible.

5. References
[1] Bancila I 1958 Geology of the Eastern Carpathians. Scientific Publishing House, Bucharest
[2] Bolis B, Di Renzo A 1959 Road pavements. Publisher Ulrico Hoelpi Milan
[3] Cososchi B, Gugiuman Gh, Vlad V N 1981, Road Laboratory Guide, „Gheorghe Asachi” Polytechnic Institute from Iași, Faculty of Constructions – course guide

[4] Grasu C, Catana C, Grinea D, Ionesi L 1975 Geological and geochemical considerations on Oligocene bituminous rocks between Pârâul Calu and Tazlau. Work. State. "Oak", geol. - grogr., VI, Pângarați

[5] Matasaru Tr, Craus I, Dorobanțu St 1966, Drumuri. Ed Tehnica București

[6] Vrtis C M 2013 Creating a performance – based asphalt mix design to incorporate Uinta basin oil sands. Master thesis, Department of Civil and Environmental Engineering, The University of Utah

[7] Nițulescu I, Spiroiu P, David M, Butucescu D 1977 Contribuții la cunoașterea compoziției și structurii șisturilor bituminoase de la Anina și a transformarilor suferite în procesele termice în vederea valorificării lor complexe (in Romanian), D.S. Inst. de Geol. și Geofiz., București

[8] Ionița G 2017 The physical – mechanical properties of asphalt mixtures containing bituminous sand, The XXV Intern Scientific Conf “trans&MOTAUTO’17”, year XI, iss 5/2017, ISSN 1313-0226, Technical Union of Mechanical Engineering „INDUSTRY 4.0”, Bulgaria, pp 246 – 249

[9] Ionița G 2017 Bituminous sand road asphalt pavement, Bulletin of the Polytechnic Institute of Jassy, Construction, Architecture Section, vol. 63 (67), Nr 3, ISSN 2068-4762, pp 55 – 63