Sand equivalent and methylene blue value of aggregates for highway engineering

Evangelos Manthos
Aristotle University of Thessaloniki

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A. NIKOLAIDES*, E. MANTHOS, M. SARAFIDOU
Aristotle University of Thessaloniki, Department of Civil Engineering
54006 Thessaloniki, Greece

SAND EQUIVALENT AND METHYLENE BLUE VALUE OF AGGREGATES FOR HIGHWAY ENGINEERING

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The suitability of the aggregates for the construction of base course and asphalt layers of a pavement is determined by various parameters-properties. One of these properties is aggregate cleanliness. The two basic tests that determine the cleanliness of the aggregates are the sand equivalent test and the methylene blue test. In the present paper, firstly, a reference is being made to the two tests and to the limits used by international standards. Secondly, relevant results are presented from a significant number of limestone and 'non-limestone' aggregates. From the results obtained it is concluded that some aggregates with acceptable sand equivalent value do not satisfy the methylene blue requirement. Similarly, aggregates that do not satisfy the sand equivalent requirement are suitable to be used in highway engineering.

Key words: Aggregates, Sand Equivalent, Methylene Blue Test

1. INTRODUCTION

One of the basic parameters for achieving proper pavement construction is the suitability of the aggregates used in the unbound and bitumen bound layers. This suitability depends on various parameters related to physical and mechanical characteristics of the aggregates. One basic parameter is their cleanliness from clay materials.

*Corresponding author. Tel.: 0030-2310-995759; fax: 0030-2310-995764
E-mail address: anik@civil.auth.gr (A. Nikolaides)

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It is important to determine whether the clay materials are harmful to the performance of the pavement layers. The harmful materials are susceptible to moisture i.e. they absorb moisture causing swelling and debonding of the bitumen from the aggregates. Thus, the question raised is: How to detect the presence of clay particles and how to determine whether they are harmful or not.

The laboratory tests that give the answers to the above are the well known sand equivalent test and the less well known methylene blue test.

In this paper, after a brief explanation of swelling-expansion mechanism, a reference is made to the two tests and to the limits proposed and used by international standards. Secondly, relevant useful results are presented from a significant number of limestone and 'non-limestone' aggregates sampled from Central and Northern Greece.

2. SWELLING – EXPANSION OF CLAY

In highway engineering the general term - clay is used to describe very small particles that may have a detrimental effect on the performance of unbound and bound materials. In terms of grain size, clay refers to particles less than 2μm in diameter. The structures of the common clay minerals are made up of combinations of two simple structural units, the silicon tetrahedron and the aluminum or magnesium octahedron [1, 2].

Clay materials occur in small particle sizes and their unit cells ordinarily have a residual negative charge in their surface. When clay particles are placed in water, the space between the particles is taken over by water molecules, which behave as dipoles with a negative pole, the oxygen atom a positive pole and the two atoms of hydrogen. At the same time the presence of the negatively charged clay materials and the dipoles of water, leads to the development of electro molecular forces which attract and hold the dipoles of water. The charged surface and the distributed charge around the particles are together termed the diffuse double layer [2].

Clay particles are multi layer formations. The distance between layers and the presence of free cations in the interlayer space are the main reasons for the swelling or not of clay. These two characteristics vary between the clay minerals. The presence of free cations results in the absorption of water dipoles to the interlayer, so that chemical balance is achieved. When the distance between the layers is small, the attractive forces among the layers are powerful and overwin the absorption of water, so swelling does not occur. On the contrary, when the distance between the layers is significant, the interlayer bonds are weak. In this case, the absorption of water in the interlayer occurs and continues until the bonds fail. At that time, the clay particles are separated and rearranged and swelling is observed. The magnitude of the phenomenon depends on other factors too which can act individually or together and magnify or decrease the major mechanisms that are mentioned above. Such factors are the density of the surface charge, the type and the valence of the cations, the concentration of the electrolytes and the dielectric constant [2].

As it is concluded from the above, swelling depends on the type of clay mineral. Swelling minerals are considered to be the minerals of the smectite group with a major representative -the montmorillonite. Non swelling clay minerals are : the pyrophyllite, the margarite and the illite.

3. LABORATORY TESTS

3.1 Sand equivalent test

The objective of the test is to quickly evaluate the relative portion of clay in the sand that is to be used in unbound and bituminous bound layers. A low value of sand equivalent characterize the fine aggregate as "dirty" and indicates that possibly the clay materials are harmful.

The test is carried out on the 0/2mm fraction in fine aggregates according to the EN 933-8 specification [3]. From a representative portion of sand, a sample of 120gr is taken and is poured into a plastic graduated cylinder. Then, a washing solution of calcium chloride is added into the cylinder, until it reaches the height of almost 100 mm. The cylinder is left for 10±1 min to soak the test sample. At the end of the 10 min period the cylinder is sealed and shaked for a period of 30±1 sec. The shaking can be done by hand or by a shaker. After the shaking the cylinder is replaced in an upright vertical position and more solution of calcium chloride is added. The adding stops when the level of the liquid reaches the upper mark of the 380±0.25 mm. The cylinder is left to settle, without disturbance and free from vibration for 20±0.25min. At the end of this period, the heights of the sand (h_s) and clay (h_c) are measured. The sand equivalent value is determined by equation 1:

$$SE = (h_s / h_c)x100 \tag{1}$$

For a specific aggregate, two cylinders are used. The sand equivalent value is calculated as the average of the values of the two cylinders and is recorded to the nearest whole number.

The minimum values of sand equivalent specified by various specifications for unbound and bound layers are given in Table 1.

Table 1. Specification limit values for the sand equivalent

<table>
<tr><th>Aggregates for:</th><th>Greek Specs [5]</th><th>Revised Greek Specifications[a]</th><th>ASTM 2940
ASTM 3515</th><th>French specs.[7]</th></tr>
<tr><td>Bituminous mixtures for wearing courses</td><td>≥55</td><td rowspan="2">≥55</td><td>≥50</td><td>≥60</td></tr>
<tr><td>Bituminous base course</td><td>≥50</td><td>≥50</td><td>≥50</td></tr>
<tr><td>Unbound base course</td><td>≥50</td><td rowspan="2">≥40</td><td>≥35</td><td rowspan="2">≥40 or ≥50 or ≥60</td></tr>
<tr><td>Unbound sub-base course</td><td>≥40</td><td>≥35 (≥30)</td></tr>
<tr><td>Cold bituminous mixtures
- for wearing course
- for binder or base layer</td><td>
≥55
≥45</td><td>-</td><td>-</td><td>≥50</td></tr>
<tr><td>Surface dressing</td><td>≥55</td><td>-</td><td>-</td><td>≥60</td></tr>
</table>

(a) To be authorized [6]

3.2 Methylene blue test

The methylene blue test is executed in order to determine whether the clay minerals are active and harmful. The active clay materials expand, depending on the moisture content. Methylene blue test is the only test that gives this information accurately and quickly. The test is carried out according to the EN 933-09 [4].

An appropriate quantity of the aggregate is dried (200g-210g of the 0/2mm fraction or 30±0.1g of the 0/0.125 mm fraction) and placed in a beaker with 500 ml of demineralized water. The mixture is stirred for 5 min. At the end of the 5 minute period, 5 ml of the methylene blue dye solution is added. The new mixture is stirred again for 1 min. Then, a “stain” test is executed. The stain test is carried out by dipping a glass rod into the mixture and then allowing a drop of the mixture to fall onto a filter paper. If the test portion has absorbed all the dye, the drop on the filter paper appears as a spot of blue stained grains surrounded by a colorless halo. The test is considered positive when the central spot is surrounded by a light blue halo, with a thickness of almost 1 mm. If after the addition of the 5 ml of the methylene blue dye solution a halo does not appear, 5 ml of the dye solution is added, the mixture is stirred again and a test spot is carried out. If again, a halo does not appear the procedure is repeated, in exactly the same way, until the halo appears. The halo must remain visible for 5 min in order to consider that the test has been finished. If the halo disappears at the first 4 minutes another 5 ml of the dye solution is added. If it disappears during the fifth minute, only 2 ml of the dye solution is added. After the test is finished, the total volume V_i of the methylene blue dye solution which was used for the formation of the halo (retained visible for 5 minutes) is recorded to the nearest 1 ml.

The Methylene blue value MB, recorded in grams of dye solution per kilograms of the aggregate of the 0/2mm fraction is given by equation 2:

$$MB = (V_i / M_i) x10 \quad \text{g/kg} \tag{2}$$

Where M_i is the mass of the sample in grams and V_i is the total volume added in milliliters

In case that the 0/0.125mm fraction is used the result is recorded as MB_F. In every case, the methylene blue value is recorded to the nearest 0.1 g of the dye solution per kilogram of aggregate fraction.

At present, there aren't many countries that have included this test as an acceptance requirement in their specifications. France, USA and now Greece are some of the few countries that have implemented this test in their National specifications. The limit values required are as shown in Table 2.

Table 2. Permissible Methylene Blue values according to international specifications

Aggregates for:	French specifications NF XP P 18-540 [7]		ISSA[(a)] TB 145	Greek Specifications (EOAE) [(b)] [9]	
	MB	MB_F	MB_F [(c)]	MB	MB_F
Unbound and bituminous bound layers	≤2.0 whenS.E.<60 ≤2.5 whenS.E.<50 ≤3.0 when S.E.<40	≤10	-	≤3.0	≤10
Wearing courses	≤2	≤10	-	-	≤10
Concrete	≤1	≤10	-	-	-
Railway works	≤1 ή ≤2	-	-	-	-
Micro-surfacing (slurry seal) and cold mixtures	≤2	≤10	≤10 or ≤7	-	≤10

(a) ISSA= International Slurry Seal Association, USA [8]
(b) EOAE= Egnatia Odos S.A. (Major State owned construction company)
(c) The test is performed on a 0/0.075 aggregate fraction

4. AGGREGATES TESTED

The aggregates examined in this paper come from 15 quarries, situated around Central and Northern Greece. In addition to the above, one steel slug was also tested. Eight of the samples were limestone aggregates and seven were non limestone aggregates suitable for wearing courses with respect to their hardness. Table 3 shows the region of the aggregate quarry and the corresponding numbering assigned. It is mentioned, that samples 15-1 to 15-7 from quarry No. 15 are collected from stockpiles having different storage period while samples 15-8 to 15-14 are collected from constructed unbound layers. The reason for this was the fact that early sand equivalent tests showed a changing behavior in sand equivalent value with sampling time after crushing.

Prior to sand equivalent and methylene blue testing, all samples except No. 15, an analysis of particle size distribution was made, considering that samples belonged to the sand fraction. The particle size gradations for the limestone aggregates are shown in Figure 1 and for the non limestone aggregates and the slug in Figure 2. The gradation envelops shown on both figures are the recommended limits of the sand fraction for producing asphalt concrete.

As it can be seen, the particle size distribution varies among sand from different quarries. Variability was also observed on the percentage of the filler content. The percentage of the filler content varied between 8.5% to18.9%, for the limestone aggregates, and 4.5% to 12.5%, for the non limestone aggregates.

Table 3. Regions of aggregate origin and notationification of quarries

Limestone aggregates		Non limestone aggregates	
Region of origin	Notification	Region of origin	Notification
Evros A, Northern Greece	1	Evros, Northern Greece	9
Thessaloniki, Northern Greece	2-1 & 2-2	Volos, Central Greece	10
Kavala, Northern Greece	3	Ioannina, Central Greece	11
Evros B, Northern Greece	4	Grevena, Northern Greece	12
Ioannina A, Central Greece	5	Kilkis, Northern Greece	13
Ioannina B, Central Greece	6	Pella, Northern Greece	14
Evros C, Northern Greece	7	Thessaloniki, Northern Greece	15-1 to 15-7 and 15-8 to 15-14
Ioannina C, Central Greece	8	Steel slug, Northern Greece	16

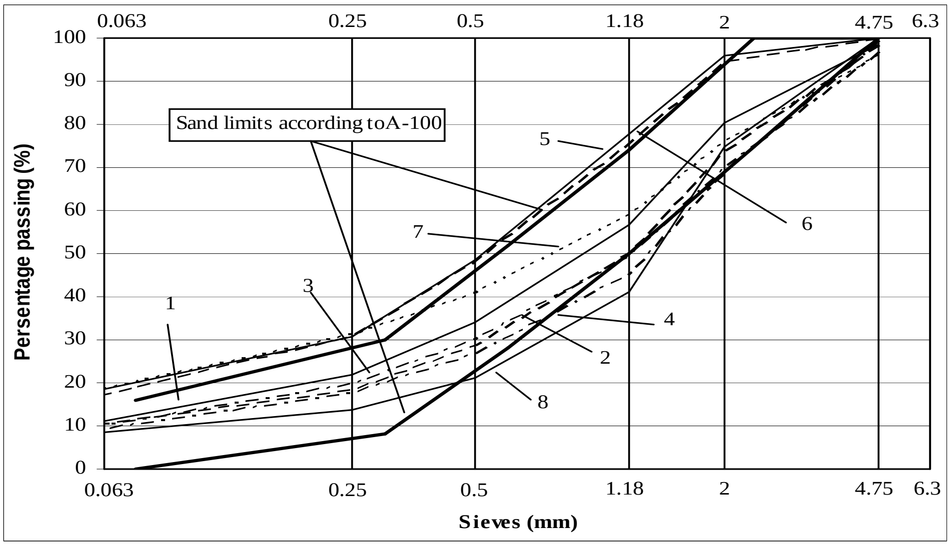

Figure 1 Sieve analysis of limestone aggregates (sand)

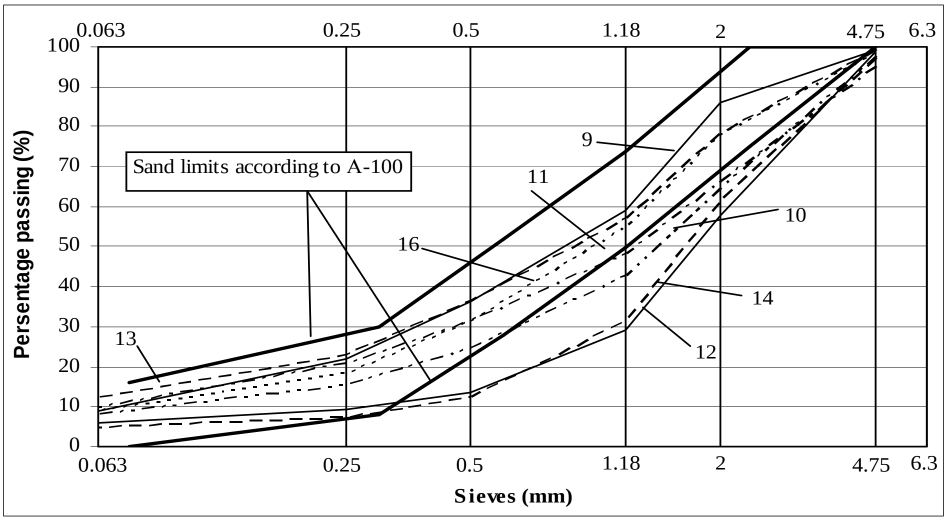

Figure 2 Sieve analysis for non limestone aggregates (sand)

5. LABORATORY RESULTS

The results obtained from the sand equivalent (SE) and methylene blue (MB) tests carried out are summarized in Table 4.

Table 4. Sand equivalent and methylene blue results

Limestone aggregates				Non limestone aggregate			
Quarry	S.E	MB	MB_F	Quarry	S.E	MB	MB_F
1	68	0.5	1.7	9	54	11.3	16.7
2-1	69	0.5	3.3	10	43	3.8	11.7
2-2	74	0.3	2.6	11	59	1.3	3.3
3	76	0.3	1.7	12	81	1.8	6.7
4	79	1.3	3.3	13	55	2.3	5.0
5	58	0.5	1.7	14	90	2.8	8.3
6	61	0.8	3.3	15-1 to 15-7	45, 40, 30, 41, 34, 36, 40	1.8, 1.8, 2.0, 1.8, 1.8, 1.8, 0.8	5.0, 5.0, 5.6, 5.0, 5.0, 5.0, 3.3,
7	70	0.3	1.7	15-8 to 15-14	22, 22, 23, 24, 22, 49, 48	2.5, 2.6, 2.7, 2.2, 1.9, 1.9, 2.0	5.0, 6.1, 6.7, 5.0, 5.0, 5.0, 5.0
8	80	0.3	1.7	16	77	0.3	1.7

6. DISCUSSION

6.1 Sand Equivalent Test

the sand equivalent values of the limestone aggregates tested varied from 58 to 80. Similarly, for the non-limestone aggregates the sand equivalent values varied from 22 to 90 respectively. According to the Greek Specifications all limestone aggregates tested are suitable for use in highway engineering.

As far as the non-limestone aggregates are concerned, only some of them are suitable for unbound and bituminous bound layers. In particular, only five out of eight materials examined, could be considered suitable for highway engineering according to sand equivalent criterion. Among the rest of the non limestone materials, the aggregate from quarry No. 10 is suitable only for sub-base layer and No. 9 is suitable for both unbound layers, but not for bituminous mixtures. Aggregate No. 15, because of its low and variable values obtained, is not acceptable neither for unbound nor bituminous bound layers.

6.2 Methylene Blue Test

The methylene blue values (MB) of the limestone aggregates varied from 0.3 to 1.3, when fraction 0/2mm was used. Similarly, the methylene blue values, when fraction 0/0.125mm was used (MB_F), varied from 1.7 to 3.3.

For the non-limestone aggregates tested, the methylene blue values (MB) varied from 0.3 to 11.3 when 0/2mm fraction was used and the MB_F from 1.7 to 16.7 when fraction 0/0.125mm was used.

According to French Standards and the draft Greek Specifications, all limestone and non-limestone aggregates, apart from No. 9 and No. 10, are suitable for both unbound and bituminous bound layers. The aggregates from quarries No. 9 and No. 10 are thought to be unsuitable as they contain harmful clay materials (swelling).

It is worth noticing that the aggregates from quarries No. 9 and No. 10 were found to be suitable if only the sand equivalent criterion was taken into consideration. On the contrary, the aggregate from quarry No. 15 was found unsuitable according to the sand equivalent criterion, alone. However, the aggregates from this quarry, despite its low to very low sand equivalent values are suitable according to the methylene blue criterion. This is because although they contain excessive amount of very fine particles ($\leq 2\mu m$), the particles are not active and harmful to pavement construction.

According to the above mentioned facts it is concluded that the application of the sand equivalent criterion alone is not safe. It could lead to the acceptance of an unsuitable aggregate or to the rejection of a suitable aggregate. The first affects the quality of works while the second affects the economy of the construction and the environmental intrusion. When additionally taking into considerationthe methylene blue criterion, proper use of natural materials is achieved. This reflects the economy of the construction cost and the protection of the environment. It must be remembered that hard and durable materials, suitable for wearing courses, in particular, are scarce in nature.

7. CORRELATION BETWEEN THE SAND EQUIVALENT AND METHYLENE BLUE VALUES

The results of sand equivalent and methylene blue values (MB_F) obtained are plotted in Figure 3.

When examining the relation between the two measures it was found that there is no correlation. The same applies if the results obtained from fraction 0/2mm were considered. This was more or less expected due to the different nature of the two tests.

What could be said for certain is that limestone aggregates with acceptable sand equivalent value, they surely satisfy the methylene blue criterion.

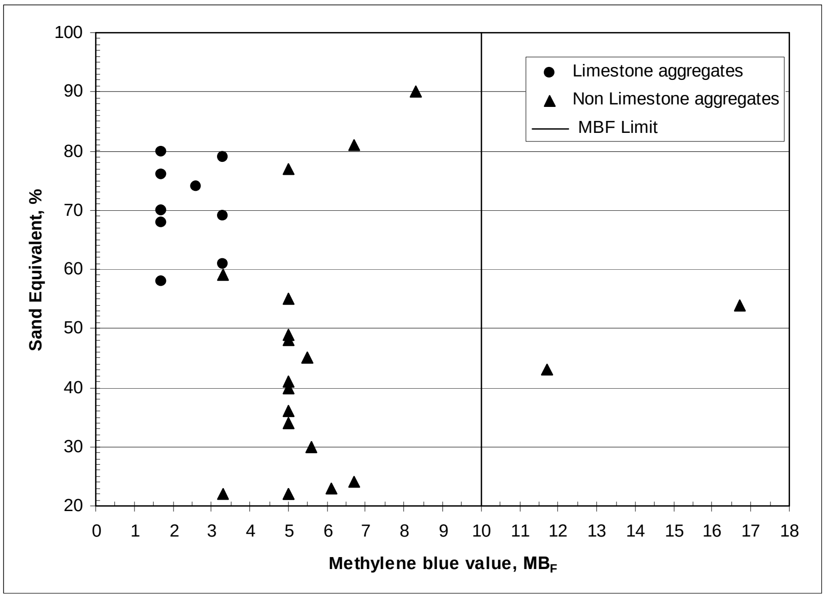

Figure 3 Sand equivalent and Methylene blue results

However, this is not the case for hard and durable non-limestone aggregates. There are cases where although the sand equivalent criterion is satisfied, the aggregates may contain harmful clay materials and hence not satisfying the methylene blue criterion. Therefore, it can be said that for non-limestone aggregates both tests must be carried out and both criteria must be applied and the acceptance decision should be based on the methylene blue criterion.

8. CONCLUSIONS

From the results obtained in this study the conclusions are as follows:

1. The limestone aggregates produced from crushing clean parent rocks posses high sand equivalent values.
2. The sand equivalent test is not a reliable test to determine whether or not any aggregate material is suitable for pavement construction.
3. For the determination of suitability of a limestone aggregate, if sand equivalent criterion is satisfied, it is not necessary to perform the methylene blue test.
4. As for the determination of suitability of a non limestone aggregate, both tests must be performed and in case the sand equivalent criterion is not sat-

isfied the acceptance or rejection should be based on the methylene blue criterion.

5. Depending on which fraction of material is preferable to be used in the methylene blue test, there seems to be no important difference, since the results in both cases provide the same information. However, using the fraction 0/0.125mm, smaller dispersion of the results and better repeatability were observed.
6. Based on the finding of (5) it is recommended to use the fraction 0/0.125mm, when the filler content in the aggregate mixture is ≥3%. If the filler content in the aggregate mixture is <3%, fraction 0/2mm may be used to run the methylene blue test.
7. Among the quarries examined it was observed that there is a big variability in relation to the aggregate gradation of the produced sands. This is not a convenient situation for a highway engineer.
8. As expected the filler content in the limestone sands grows constantly as opposed to the filler content in the non limestone sands.

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