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EVALUATION OF CRETACEOUS LIMESTONES FROM THE AITOLOAKARNANIA PROVINCE (WESTERN GREECE) FOR THEIR USE AS ROAD AGGREGATES IN TERMS OF THEIR CONTENT IN SWELLING CLAY MINERALS

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Abstract

The aim of this paper is to assess the suitability of a significant number of carbonate rocks from the Aitoloakarnania province (Western Greece) for their use as road construction aggregates, in terms of their content in swelling clay minerals. The study focuses on Cretaceous limestones from the Olonos-Pindos zone. Detailed petrographic analysis and the sand equivalent and methylene blue tests are carried out, in order to estimate the quantity of swelling clay minerals in aggregate particles. Special emphasis is given on correlating the results of the sand equivalent and methylene blue tests, as well as on the relationships between these engineering parameters and the petrographic data. The results are evaluated in accordance with the Greek and International suitability Standards for road construction aggregates. Although the results of the sand equivalent test indicate the suitability of the studied samples, the results of the methylene blue test imply that they are not suitable for use in all applications of road construction. Hence, it is proved that the methylene blue test is of fundamental importance for the determination of the lithotypes which are suitable for use as road aggregates in various applications.

Key words: *Carbonate rocks, Olonos-Pindos zone, aggregates, sand equivalent, methylene blue.*

Περίληψη

Σκοπός της εργασίας αυτής είναι η εκτίμηση της καταλληλότητας ενός σημαντικού αριθμού ανθρακικών πετρωμάτων από το Νομό Αιτ/νίας (Δυτική Ελλάδα) για χρήση τους ως αδρανή υλικά σε έργα οδοποιίας, ως προς την περιεκτικότητά τους σε διογκούμενα αργιλικά ορυκτά. Η μελέτη επικεντρώνεται σε Κρητιδικούς ασβεστόλιθους από τη ζώνη Ωλονού–Πίνδου. Πραγματοποιήθηκε λεπτομερής πετρογραφική εξέταση και επιπλέον προσδιορίστηκαν οι δοκιμές ισοδύναμου άμμου και μπλε του μεθυλενίου, έτσι ώστε να εκτιμηθεί η περιεκτικότητα των υπό μελέτη αδρανών σε αργιλικά ορυκτά. Ιδιαίτερη έμφαση δόθηκε στον προσδιορισμό των συσχετίσεων μεταξύ των δοκιμών ισοδύναμου άμμου και μπλε του μεθυλενίου, καθώς και στη σύνδεση των αποτελεσμάτων των δοκιμών αυτών με τις πετρογραφικές παραμέτρους. Οι τιμές των υπό μελέτη ασβεστόλιθων αξιολογήθηκαν με βάση τις ελληνικές και διεθνείς προδιαγραφές, στις

οποίες αναφέρονται οι τυπικές αποδεκτές τιμές όσον αφορά στην καταλληλότητά τους για χρήση ως αδρανή υλικά οδοποιίας. Παρότι τα αποτελέσματα της δοκιμής ισοδύναμου άμμου υποδεικνύουν την καταλληλότητα των υπό μελέτη δειγμάτων, από τη δοκιμή μπλε του μεθυλενίου προκύπτει ότι αυτά δεν είναι κατάλληλα για χρήση τους σε όλες τις εφαρμογές οδοποιίας. Συνεπώς, προκύπτει ότι η δοκιμή μπλε του μεθυλενίου είναι εξαιρετικής σημασίας για τον προσδιορισμό εκείνων των πετρωμάτων που είναι κατάλληλα για χρήση τους στις διάφορες εφαρμογές οδοποιίας.
Λέξεις κλειδιά: *Ανθρακικά πετρώματα, ζώνη Ωλονού–Πίνδου, αδρανή υλικά, ισοδύναμο άμμου, μπλε του μεθυλενίου.*

1. Introduction

The increasing demand for crushed rock aggregates in various applications and especially the requirement for hard aggregates in numerous infrastructure works of Greece, have increased the necessity for the detection of carbonate rocks which are suitable for the production of aggregates used for: bases and sub-bases, improvement layers, bituminous mixtures, concrete and embankments. The suitability of aggregate materials depends on their various physicomechanical properties, as well as on their content in clay minerals. In terms of grain size, clay refers to particles less than 2 μm in diameter, while clay minerals are hydrous aluminium phyllosilicates. Swelling clay minerals are considered to be the minerals of the smectite group. The latter absorb water and tend to swell, causing significant problems in the unbound road layers. Clay minerals also reduce the cohesion of bituminous mixtures, due to the fact that they destroy the adhesion between asphalt and aggregate particles (Smith and Collis, 2001; Nikolaides *et al*., 2007).

The sand equivalent test is used for the determination of clay-like fines in aggregates; however the methylene blue test is applied to obtain an assessment of the quantity of swelling clay minerals of the smectite type in a sample of aggregate. These tests are carried out in aggregates used in both unbound and bound road layers. This paper investigates the suitability of 51 carbonate rock samples from the Aitoloakarnania province (Western Greece) for their use as road construction aggregates, based on their mineralogical-textural features and on the results of the sand equivalent and methylene blue tests.

2. Geological Setting

The carbonate rocks of the Olonos-Pindos zone (Aitoloakarnania province, Western Greece) (Fig. 1), cover a total area of 417 km^2. They comprise part of the lower unit (thickness more than 1000 m) of the Olonos-Pindos zone, which includes pelagic limestones of Triassic to Upper Cretaceous age. These limestones are intercalated with radiolarites of Middle Jurassic to Lower Cretaceous, while their thickness varies from 200 to 400 m (Katsikatsos, 1992; Mountrakis, 1985). The Upper Cretaceous limestones, which comprise the upper part of this lower unit, are whitish limestones, gray micritic limestones with veins filled with secondary calcite and red limestones.

The studied area (Fig. 1) and especially the Upper Cretaceous carbonate rocks cover an area of 301 km^2, which corresponds to the 72.35 % of the overall carbonate formations of the Olonos-Pindos zone. The rock slopes of the area exhibit layers up to 35 cm thick and are cut by joints with opening up to 3-4 cm. These lithologies are characterized by intense fracturing and folding.

3. Petrography

The petrographic study of the Upper Cretaceous carbonate rocks of the Olonos-Pindos zone (Aitoloakarnania province) includes the macroscopic and microscopic examination of 51 samples. These samples were collected from representative localities of quarry faces or natural slopes, with a view to be fresh and to represent the full variability of the quarry products.

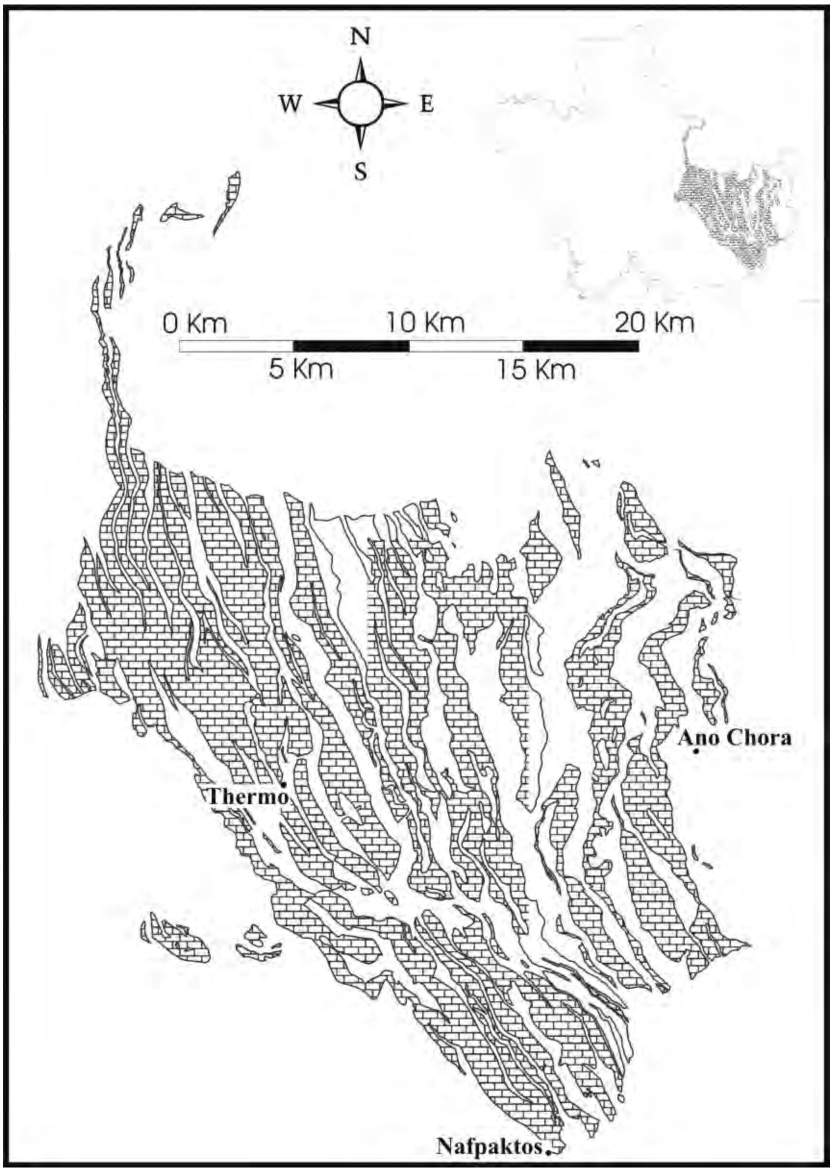

Figure 1 - Simplified geological map of the studied Upper Cretaceous limestones of the Olon-os-Pindos zone (Aitoloakarnania province).

Macroscopically, the studied rock types show a dense network of sparitic veins and their colour is whitish to gray, except for samples AT2B, AT6C, AT8C, AT9B, AT17C, AT19B, AT22B and AT42B, which have a red colour. The samples were collected based on petrographic criteria, which significantly determine their quality as aggregate material (Zarif and Tuğrul, 2003; Tsikouras *et al*., 2005; Pomonis *et al*., 2007).

The microscopic examination, which was carried out using polarized microscopy at the Research Laboratory of Minerals and Rocks, Department of Geology, University of Patras, indicated that the samples are micritic or sparitic microcrystalline limestones with veins filled with secondary calcite. Quartz crystals, as well as the phyllosilicate minerals muscovite and/or biotite are also present. The dominance of bioclasts is obvious, however many samples also contain lithoclasts, endoclasts or opaque minerals. The Upper Cretaceous limestones can be classified according to the grains/matrix ratio and their content in endoclasts, lithoclasts or bioclasts, as follows:

(a) Fossiliferous-micrites according to Folk (1962) or mudstones according to Dunham (1962) with a joint system which cuts a micritic matrix. The joints have been filled with sparitic calcite (Fig. 2a). Additionally, there is a low content of fossils in a uniform, dark coloured micritic matrix. Joints with a thickness up to 0.3 mm, which are filled with secondary, microcrystalline calcite, are also present. Moreover, areas with channel or fracture porosity are observed (Fig. 2b).

(b) Sparse biomicrites according to Folk (1962) and wackestones/packstones according to Dunham (1962). They are biomicritic limestones, which contain pieces of broken fossils, as well as sizeable and well preserved bioclasts (skeletal remains of planktonic foraminifera). In addition, the characteristic fossils of Upper Cretaceous *Calpionella* (Fig. 2c) and *Globotruncana* (Fig. 2d) occur in a micritic matrix. The joints of this lithology are filled with microcrystalline calcite, while the stylo-liths are filled with Fe-oxides and clay minerals (Fig. 2d).

(c) Packed biomicrites according to the classification scheme of Folk (1962) and packstones according to the classification scheme of Dunham (1962). These rocks contain bioclasts in a proportion of more than 50 % (Fig. 2e, f), as well as carbonate grains which are surrounded by a micritic matrix.

(d) Sorted-unsorted intrabiosparites according to Folk (1962) and grainstones according to Dunham (1962). They comprise lithoclasts, endoclasts, bioclasts (Fig. 2g, h) and opaque minerals in a sparite cement, while the micritic matrix is absent. These rocks are characterized by the presence of isometric, coarse grains of calcite, which contain abundant lithoclasts, endoclasts and fossils (Fig. 2h). The porosity is primarily channel-type, filled with sparite material. Stylolithic or fenestral porosity filled with Fe-oxides and/or clay minerals can also be observed.

4. Geometrical Properties

The suitability of the studied limestones for their use as aggregate material in highway engineering was assessed based on the results of the sand equivalent and methylene blue tests. Subsequently, the results were evaluated in accordance with the Greek and International suitability Standards for road construction aggregates (Tables 1, 2).

The sand equivalent test (SE) defines the relative proportions of clay-like fines in aggregates passing ASTM Sieve No 4 (4.75 mm), since an excess of clays is usually detrimental to the performance of any aggregate (Hveem, 1953). This test was carried out in accordance with ASTM D2419. As can be seen in Table 3, the SE values of the studied rocks are $\geq$60%; hence they are suitable for use in base and sub-base layers, bituminous mixtures for wearing courses and cold bituminous mixtures. Specifically, the SE values range as follows:

- Fossiliferous micrites: 60 – 77 %
- Biomicrites: 60 – 79 %
- Sparites: 68 – 81 %

The methylene blue test (MB), which is applied to obtain an assessment of the quantity of swelling clay minerals of the smectite type in a sample of aggregate, was determined according to the procedure described in EN 933-9. This test was determined in the fine aggregate fraction of 0-0.125 mm (MBf), taking into account that it gives results with better repeatability (Nikolaides *et al.*, 2007). The MBf values of the studied rocks range as follows:

- Fossiliferous micrites: 8.6 – 26.6 g/kg
- Biomicrites: 6.6 – 20.0 g/kg
- Sparites: 4.3 – 9.3 g/kg

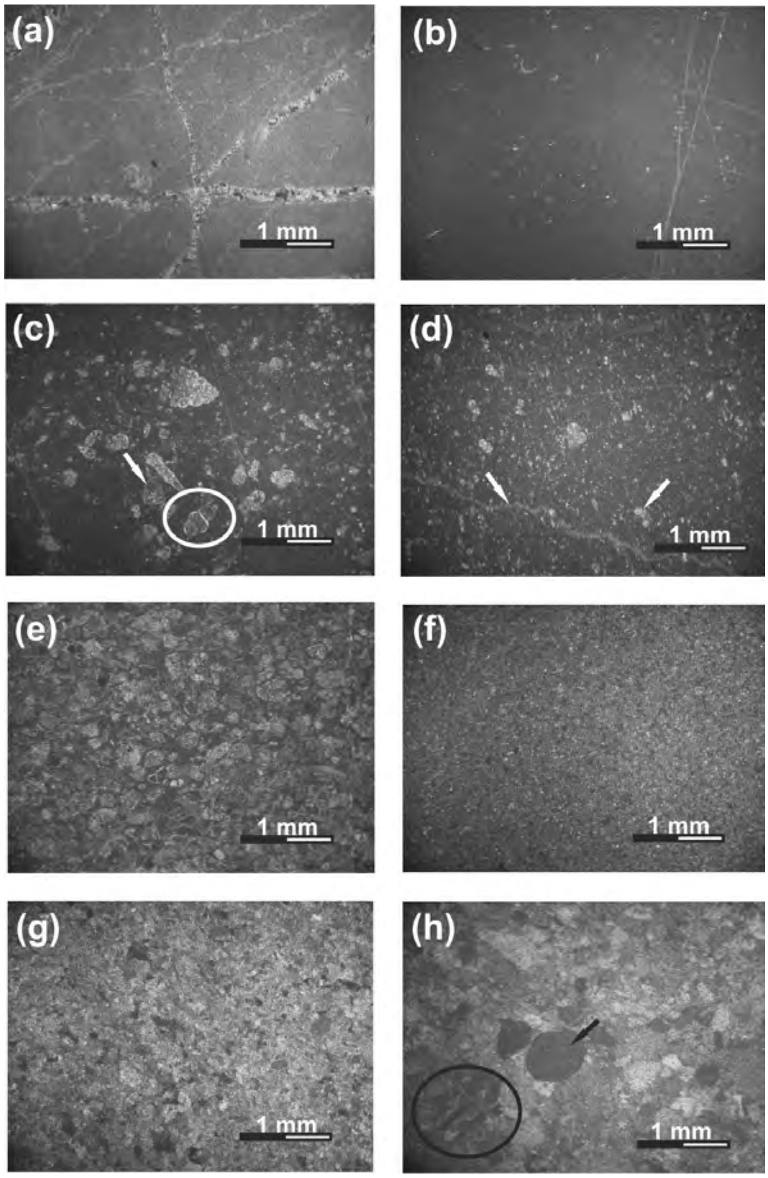

Figure 2 – (a) Fossiliferous micrite: joints filled with sparry calcite in a micritic matrix (Nicols+); (b) Fossiliferous micrite: sparsely scattered fossils in a micritic matrix with fracture porosity (Nicols+); (c) Sparse biomicrite/wackestone: pieces of broken calpionellids (arrow) and foraminifera (ellipse) (Nicols+); (d) Sparse biomicrite/wackestone: moderate participation of fossils in a rock type with fracture porosity. Fe-oxides and clay minerals fill the stylolith at the lower part of the field of view (left arrow). The right arrow shows a Globotruncana (Nicols+); (e) Packed biomicrite/packstone: abundant bioclasts (>50%) in a micritic matrix (Nicols+); (f) Packed biomicrite/packstone: bioclasts and opaque minerals (Nicols+); (g) Sorted intrabiosparite/ grainstone: participation of lithoclasts-bioclasts (Nicols+); (h) Unsorted intrabiosparite/grainstone: participation of endoclasts (arrow) and fossils (ellipse) (Nicols+).

Table 1 - Permissible sand equivalent (SE) values.

Aggregates for:	SE (%) Greek Specifications
Bituminous mixtures for wearing courses	> 55
Bituminous base courses	> 50
Unbound base courses	> 50
Unbound sub-base courses	> 40
Cold bituminous mixtures for wearing courses	> 50
Cold bituminous mixtures for base layers	> 45

Table 2 - Permissible methylene blue (MBf) values.

Aggregates for:	Specifications (MBf) NF XP P 18-540	Greek Specifications (MBf)
Base and sub-base layers	$\leq 10^{(3)}$	$\leq 10^{(1)}$
Bituminous bases	$\leq 10^{(3)}$	≤ 10
Bituminous bound layers	$\leq 10^{(3)}$	≤ 10
Wearing courses	$\leq 10^{(3)}$	≤ 10
Concrete	$\leq 10^{(3)}$	
Cold bituminous mixtures Micro-surfacing layers	≤ 7 or ≤ 8 MBf $^{(2)}$ ≤ 10 MBf $^{(2)}$	

(1) Egnatia Odos S.A.
(2) Specifications of other countries
(3) French specifications

(e.g. linear, logarithmic) and it was observed that the logarithmic model gives the higher R^2 values. As can be seen in Figure 3, different trends were calculated for fossiliferous micrites, biomicrites and sparites, which are described by the following equations:

$$SE = -12.962 \times \ln MBf + 105.79, \quad R2 = 0.5666 \quad \textit{(Fossiliferous micrites)}$$

$$SE = -9.0277 \times \ln MBf + 94.156, \quad R2 = 0.3255 \quad \textit{(Biomicrites)}$$

$$SE = -10.157 \times \ln MBf + 91.664, \quad R^2 = 0.3853 \quad \textit{(Sparites)}$$

The calculated R^2 values indicate weak to moderate negative correlations between SE and MBf. The insignificant correlations between these geometrical parameters can be attributed to the different nature of the sand equivalent and methylene blue tests.

Based on the petrographic classification according to Folk (1962) and Dunham (1962) and the dispersion of the results of the geometrical properties (Fig. 3), the studied carbonate rocks can be further grouped into:

A) Fossiliferous micrites

1st Group: Samples AT7A, AT10A and AT11A with MBf values varying between 8.6 and 9.6 g/kg and SE values ranging from 76 to 77 %.

2nd Group: Samples AT6D, AT8A, AT13A, AT13B, AT17B, AT17C, AT20B and AT42B with MBf values from 13.0 to 26.6 g/kg and SE values from 60 to 76 %. The substantially higher MBf values of the samples of this group, compared to those of the first group, are due to the clay minerals which fill the styloliths of the samples of the second group.

Table 3 - Results of the Methylene Blue and Sand Equivalent of the studied rock samples from the Olonos-Pindos zone (Aitoloakarnania province).

Sample	Rock Type	Methylene Blue (g/kg)	Sand Equivalent (%)
AT6D	Fossiliferous micrite	21.0	64
AT7A	Fossiliferous micrite	9.6	76
AT8A	Fossiliferous micrite	26.6	68
AT9B	Fossiliferous micrite	19.6	63
AT10A	Fossiliferous micrite	8.6	76
AT11A	Fossiliferous micrite	9.0	77
AT13A	Fossiliferous micrite	13.0	76
AT13B	Fossiliferous micrite	16.6	70
AT17B	Fossiliferous micrite	16.0	77
AT17C	Fossiliferous micrite	17.6	65
AT20B	Fossiliferous micrite	16.6	73
AT42B	Fossiliferous micrite	20.3	60
AT2A	Biomicrite	13.3	71
AT2B	Biomicrite	16.3	68
AT4A	Biomicrite	10.7	70
AT6A	Biomicrite	8.0	77
AT6C	Biomicrite	10.6	67
AT7B	Biomicrite	8.0	76
AT8C	Biomicrite	16.6	68
AT11C	Biomicrite	8.3	72
AT12A	Biomicrite	6.6	77
AT12B	Biomicrite	10.0	78
AT14A	Biomicrite	10.0	69
AT15A	Biomicrite	11.6	71
AT16A	Biomicrite	16.6	60
AT18B	Biomicrite	11.0	73
AT18C	Biomicrite	15.0	75
AT19A	Biomicrite	12.3	77
AT19B	Biomicrite	14.0	68
AT22A	Biomicrite	9.0	73
AT22B	Biomicrite	20.0	72
AT22C	Biomicrite	10.0	71
AT48	Biomicrite	8.3	77
AT49A	Biomicrite	10.0	79
AT49B	Biomicrite	15.0	70
AT54	Biomicrite	10.0	76
AT2C	Sparite	8.6	77
AT4B	Sparite	5.6	76
AT6B	Sparite	7.6	68
AT8B	Sparite	9.3	67
AT9A	Sparite	5.6	68
AT10B	Sparite	8.6	70
AT11B	Sparite	8.3	68
AT14B	Sparite	9.0	72
AT15B	Sparite	6.6	70
AT16B	Sparite	9.3	71
AT17A	Sparite	4.3	81
AT18A	Sparite	5.0	77
AT19C	Sparite	6.3	69
AT20A	Sparite	5.0	76
AT42A	Sparite	9.3	68

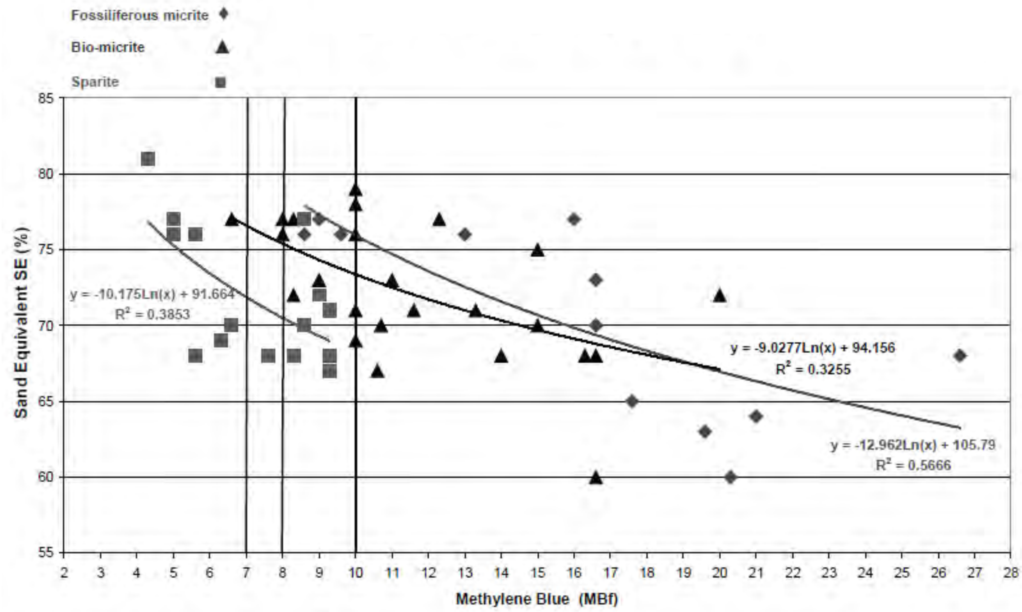

Figure 3 - Correlation between methylene blue (MBf) and sand equivalent (SE) of the studied limestones.

B) Biomicrites

1st Group: Samples AT12A, AT6A, AT11C, AT22A and AT7B whose MBf values range from 6.6 to 9.0 g/kg and SE values from 72 to 77 %.

2nd Group: Samples AT2A, AT2B, AT4A, AT6C, AT8C, AT12B, AT14A, AT14B, AT15A, AT16A, AT18B, AT18C, AT19A, AT19B, AT22B, AT48, AT49A, AT49B, AT54 and AT22C with MBf values ranging from 8.3 to 16.6 g/kg and SE values ranging from 60 to 79 %. The stylolith or channel porosity of these samples has been filled with clay material and this is considered to be the reason for their higher MBf values.

C) Sparites

1st Group: Samples AT2C, AT4B, AT17A, AT18A and AT20A with MBf varying from 4.3 to 8.6 g/kg and SE ranging from 72 and 81 %. The higher MBf value of sample AT2C compared to the other samples is attributed to its stylolithic porosity, which is filled with Fe-oxides and clay minerals.

2nd Group: Samples AT6B, AT8B, AT6C, AT9A, AT12B, AT11B, AT14B, AT15B, AT16B, AT19C and AT42A with MBf ranging between 5.6 and 9.3 g/kg and SE varying from 67 to 73 %. The higher MBf and lower SE values of these samples, compared to those of the first group, are assigned to the fact that the samples of the second group have higher percentage of stylolythic and channel porosity, filled with clay material.

5. Discussion

The petrographic features, as well as the degree of tectonic deformation and porosity of rocks are determinative parameters for their quality as aggregate material (Hartley, 1974; Kazi and Al-Mansour, 1980; Al-Jassar and Hawkins, 1991; Smith and Collis, 2001; Jensen *et al*., 2010). Additionally, the geometrical properties of aggregates play an important role in the estimation of aggregate performance in-service (Nikolaides *et al*., 2007; Rigopoulos *et al*., 2013). The results of this study bring new details to the understanding of the interrelations between the sand equivalent test, the methylene blue test and the petrographic characteristics of carbonate rocks. The suitability of the studied samples for their use as aggregates in road construction is also assessed.

The petrographic examination indicated that the carbonate rocks of this study can be grouped into: (a) fossiliferous micrites, (b) biomicrites, and (c) sparites. The geometrical properties of these lithotypes seem to be highly controlled by their microscopic features. Those samples which have stylolithic and/or channel porosity, filled with clay material, tend to have lower sand equivalent and higher methylene blue values, implying the interdependence among petrography and engineering parameters. Similar relationships have been referred by various researchers (e.g. Miskovsky *et al*., 2004; Kondelchuk and Miskovsky, 2008; Rigopoulos *et al*., 2013).

Regarding the correlation between sand equivalent and methylene blue, regression analysis indicated that there is no significant relationship between these geometrical parameters due to the different nature of the two tests. Similar results have also been referred by Nikolaides *et al*. (2007).

The values of the sand equivalent test for the studied Upper Cretaceous limestones are ≥60%; hence they are all considered suitable for use in base and sub-base layers, bituminous mixtures for wearing courses and cold bituminous mixtures. However, the sand equivalent test defines the relative proportions of clay-like fines in aggregates; thus the results of the methylene blue test, which determines the quantity of swelling clay minerals, should also be taken into consideration. Samples AT2C, AT14B, AT16B, AT10B, AT42A, AT8B, AT11B, AT11C, AT14B, AT7A, AT14B, AT10A, AT22A, AT7A and AT11A have MBf values ≤10 g/kg and ≥8 g/kg, so they are suitable for use in base and sub-base road layers and bituminous mixtures for wearing courses. Samples AT4B, AT6A, AT6B, AT7B, AT9A, AT12A, AT15B, AT17A, AT18A, AT19C and AT20A, which show MBf values ≤8 g/kg, can also be used as aggregates in cold bituminous mixtures. On the other hand, samples AT12B, AT14A, AT22C, AT49A and AT54 are near the MBf limit of 10 g/kg and exhibit relatively high sand equivalent values (SE: 69-79%). The rest of the samples (AT2A, AT2B, AT4A, AT6C, AT6D, AT8A, AT8C, AT9B, AT13A, AT13B, AT15A, AT16A, AT17B, AT17C, AT18B, AT18C, AT19A, AT19B, AT22B, ATA22B, AT42B and AT49B), whose MBf values range from 10.6 to 26.6 g/kg, are unsuitable for use in road construction due to the high proportion of swelling clay minerals in their porosity.

6. Conclusions

The carbonate lithotypes of this study can be grouped into: (a) fossiliferous micrites, (b) biomicrites, and (c) sparites. Their geometrical parameters seem to be significantly controlled by their mineralogical and textural characteristics. The samples which have stylolithic and/or channel porosity, filled with clay material, tend to have lower sand equivalent and higher methylene blue values. This indicates that the knowledge of the petrographic features of rocks is of great importance for the estimation of their engineering behaviour.

There is insignificant relationship between the results of the sand equivalent and methylene blue tests, which is assigned to the different nature of these two geometrical properties.

The results of the sand equivalent test indicate that the studied samples are suitable for use as aggregates in base and sub-base layers, bituminous mixtures for wearing courses and cold bituminous mixtures; however the results of the methylene blue test imply that a number of the studied limestones are not suitable for use in road construction. Thus, it is proved that the methylene blue test is of fundamental significance for the determination of the lithotypes which are suitable for use as road aggregates in various applications.

7. Acknowledgements

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