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Determination of Methylene Blue Index in Silica Sand–Clay Mixtures Using ASTM C837-09

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ABSTRAK

Silica sand is a material used in the cement industry, while clay can serve as a raw material component that influences the characteristics of a mixture. This study examines mixtures of silica sand and clay, prepared with varying clay content, to analyze the relationship between the proportion of clay and the methylene blue index value. Testing was conducted in accordance with the procedures outlined in ASTM C837-09. Samples were prepared with ten different clay concentrations, ranging from 1% to 10%. Each sample was mixed with distilled water and tested using a methylene blue solution until a blue halo appeared on the filter paper, signifying the test's endpoint. The results indicate that the methylene blue index value increases from 0.15 at a 1% clay content to 0.70 at a 10% clay content. A linear regression analysis yielded a coefficient of determination (R^2) of 0.9519, demonstrating that the linear model accounts for 95.19% of the variation in the test values within the analyzed data. These findings suggest a strong linear relationship between clay content and the methylene blue index value within the tested range. Nevertheless, further testing—incorporating comparative methods and repeated trials—is required to evaluate the accuracy of this method in estimating clay content.

Kata Kunci:

clay content, pasir silika, *clay*, metilen biru, ASTM C837-09.

Pasir silika merupakan salah satu bahan yang digunakan dalam industri semen, sedangkan clay dapat menjadi bagian dari bahan baku dan memengaruhi karakteristik campuran. Penelitian ini menggunakan campuran pasir silika dan clay yang dibuat secara sengaja dengan variasi kadar clay untuk menganalisis hubungan antara proporsi clay dan nilai indeks metilen biru. Pengujian dilakukan menggunakan prosedur yang mengacu pada ASTM C837-09. Sampel disiapkan dengan sepuluh variasi kadar clay, yaitu 1% hingga 10%. Setiap sampel dicampurkan dengan akuades dan diuji menggunakan larutan metilen biru hingga terbentuk halo biru pada kertas saring sebagai penanda titik akhir pengujian. Hasil pengujian menunjukkan bahwa nilai indeks metilen biru meningkat dari 0,15 pada kadar clay 1% menjadi 0,70 pada kadar clay 10%. Analisis regresi linier menghasilkan koefisien determinasi (R^2) sebesar 0,9519, yang menunjukkan bahwa model linier dapat menjelaskan 95,19% variasi nilai pengujian pada data yang dianalisis. Hasil ini menunjukkan adanya hubungan linier yang kuat antara kadar clay dan nilai indeks metilen biru dalam rentang pengujian. Namun, pengujian lebih lanjut menggunakan metode

pembanding dan pengulangan diperlukan untuk mengevaluasi keakuratan metode dalam memperkirakan kadar clay.

INTRODUCTION

The cement industry requires quality control of raw materials to ensure the consistency of the composition used in the clinker production process. Clay serves as a raw material that contributes silica (SiO_2) and alumina (Al_2O_3) to cement manufacturing, while the characteristics of its constituent minerals can influence the material's properties. Therefore, the characteristics of clay-bearing materials must be evaluated in relation to their intended use (Singh, 2022).

One method used to evaluate the characteristics of clay is the Methylene Blue Index (MBI) test. ASTM C837-09 (reapproved 2024) establishes a procedure for measuring the adsorption of the dye methylene blue by clay, expressing the result as the Methylene Blue Index. While the Methylene Blue Index value relates to specific clay characteristics—such as cation exchange capacity and dry bonding strength—the results cannot be directly attributed to a single, simply measured clay property (ASTM, 2009)

Research by Miyoshi et al., (2024) explores the use of methylene blue adsorption to estimate the bentonite content in bentonite–sand mixtures. The study demonstrates that methylene blue adsorption measurements can be used to estimate the bentonite content in mixtures with a predetermined composition, employing a colorimetric method adapted from JIS Z 2451:2019 (Miyoshi et al., 2024). Other research, by Bijang et al., (2024), evaluates methylene blue adsorption in natural clay from Ouw, while Hamad et al., (2024) examine the use of bentonite for the removal of methylene blue from water.

Although previous research has addressed the adsorption of methylene blue by clay and its application in estimating the bentonite content within sand mixtures, the material characteristics and testing procedures employed in those studies differ from those used in this research. For instance, (Miyoshi et al., 2024) utilized bentonite–sand mixtures and a modified colorimetric method, whereas this study employs silica sand–clay mixtures with predetermined clay content levels. Consequently, this research was conducted to evaluate the relationship between the percentage of clay in silica sand–clay mixtures and the response obtained from the methylene blue test, based on the specific laboratory procedures applied.

This study aims to analyze the relationship between varying clay percentages (ranging from

1% to 10%) in silica sand–clay mixtures and the results of the methylene blue test. The percentage of clay in the mixture is treated as the independent variable, while the methylene blue test result serves as the response variable. The relationship between these two variables is analyzed to determine the pattern of change in the test response as the clay percentage increases. The testing was performed in accordance with the principles of ASTM C837-09, with modifications made to the sample type and mass, water volume, the sequence of methylene blue solution addition, and the stirring duration.

METHOD

The methylene blue index test was conducted from January 21 to 26, 2026, at the Chemical Laboratory of PT Semen BOSOWA Maros. The equipment used in this experiment included a grinding device, magnetic stirrer, analytical balance, 10 mL burette, 250 mL beaker, 150-mesh sieve, stirring rod, ball stirrer, and spatula. The materials used were distilled water, clay, filter paper, methylene blue, and silica sand.

a. Sample Preparation

Approximately 500 g of silica sand and 240 g of clay were prepared as the starting materials for the research. The silica sand was ground until fine and then passed through a 150-mesh sieve to ensure a uniform particle size. The clay, meanwhile, was already in powder form with a 150-mesh particle size, so no further sieving was required. Subsequently, the silica sand and clay were weighed according to specific clay content variations—1%, 2%, 3%, 4%, 5%, 6%, 7%, 8%, 9%, and 10% of the total mixture mass. Each variation was prepared with a total mass of 100 g, consisting of: 99 g of silica sand and 1 g of clay for the 1% variation; 98 g of silica sand and 2 g of clay for the 2% variation; 97 g of silica sand and 3 g of clay for the 3% variation; 96 g of silica sand and 4 g of clay for the 4% variation; 95 g of silica sand and 5 g of clay for the 5% variation; 94 g of silica sand and 6 g of clay for the 6% variation; 93 g of silica sand and 7 g of clay for the 7% variation; 92 g of silica sand and 8 g of clay for the 8% variation; 91 g of silica sand and 9 g of clay for the 9% variation; and 90 g of silica sand and 10 g of clay for the 10% variation. The two materials were then mixed and thoroughly homogenized inside a plastic bag until a uniform blend was achieved. Each mixture was stored in a labeled container for use in the subsequent testing phase.

b. Methylene Blue Test on Silica Sand–Clay Mixtures

A 1 g portion of each homogenized silica sand and clay mixture—prepared with clay content variations of 1%, 2%, 3%, 4%, 5%, 6%, 7%, 8%, 9%, and 10%—was weighed. Each sample was placed into a beaker, combined with 100 mL of distilled water, and homogenized using a magnetic stirrer for approximately 5 minutes. Subsequently, a 1% methylene blue solution was added in 0.5 mL increments. Following each addition, the mixture was stirred with a magnetic

stirrer for about 3 minutes. To determine the test endpoint, a drop of the mixture was placed onto filter paper. The endpoint was identified by the appearance of a light-blue ring, or "halo," around the droplet that remained visible after being left to stand for approximately 1 minute. This procedure is based on the methylene blue index test principle outlined in ASTM C837-09, with modifications made to the sample mass and type, water volume, the incremental addition of the solution, and the stirring duration.

RESULTS

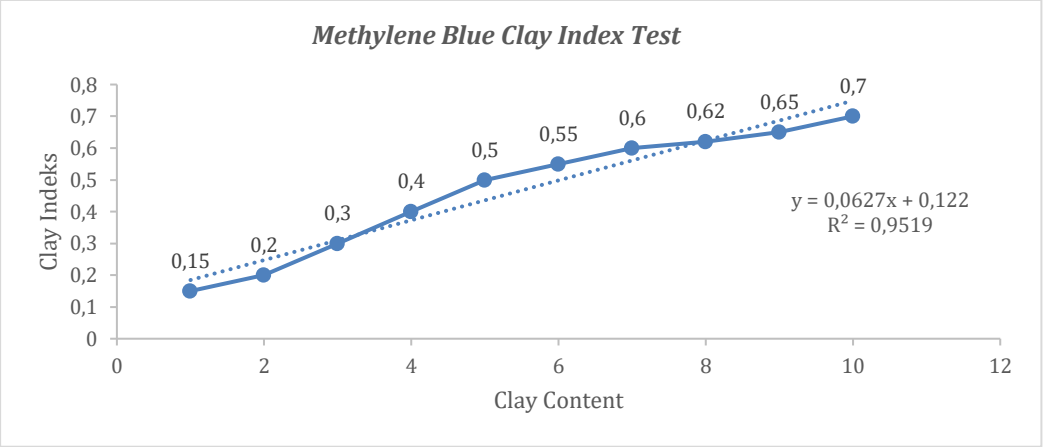
The methylene blue index (MBI) is a parameter used to evaluate the adsorption of methylene blue by clay-containing materials. Demonstrated that methylene blue adsorption can be used to estimate bentonite content in bentonite–sand mixtures. In this study, the methylene blue test was used to evaluate the adsorption response of silica sand–clay mixtures containing different clay proportions (Miyoshi et al., 2024).

Tabel 1. *Methylene Blue Clay Index Test Data*

x	y	x^2	y^2	x.y
1	0.15	1	0.0225	0.15
2	0.20	4	0.04	0.4
3	0.30	9	0.09	0.9
4	0.40	16	0.16	1.6
5	0.50	25	0.25	2.5
6	0.55	36	0.3025	3.3
7	0.60	49	0.36	4.2
8	0.62	64	0.3844	4.96
9	0.65	81	0.4225	5.85
10	0.70	100	0.490	7

The methylene blue test results showed an increase in the measured index value from 0.15 to 0.70 as the nominal clay proportion in the silica sand–clay mixtures increased from 1% to 10%. This trend indicates a positive relationship between the clay proportion and the methylene blue adsorption response under the experimental conditions. However, the increase in the measured index does not, by itself, establish the accuracy of the method for determining clay content. Further validation using an independent reference method or reference materials is required to assess its accuracy.

Figure 1. Methylene Blue Clay Index Test Graph



Linear regression analysis was used to evaluate the relationship between clay proportion (X) and the measured methylene blue index (Y) in silica sand–clay mixtures. The coefficient of determination (R^2) was 0.9519, indicating that the linear model explained 95.19% of the variation in the measured index values within the analyzed dataset. However, this result describes the fit of the regression model and does not, by itself, establish the accuracy or validity of the method for determining clay content. Nyankson et al., (2020) reported that clay materials exhibited methylene blue adsorption, with adsorption behavior affected by the properties and treatment of the clay. Therefore, the results of this study indicate a strong linear relationship between the nominal clay proportion and the measured index within the tested range, while further validation is required to establish the method's accuracy.

CONCLUSION

The results showed that increasing the clay proportion from 1% to 10% in the silica sand–clay mixtures was accompanied by an increase in the measured methylene blue index from 0.15 to 0.70. Linear regression analysis yielded an R^2 value of 0.9519, indicating a strong linear relationship between the clay proportion and the measured index values. These findings demonstrate that the methylene blue test response varied with the clay proportion in the mixtures. However, further validation is needed to determine the accuracy of the method for estimating clay content.

RECOMMENDATIONS

It is recommended that the procedure be carried out with greater precision—particularly regarding the addition of methylene blue and the determination of the titration endpoint—to ensure the results obtained are more accurate and consistent.

Allah swt, berfirman dalam QS. Al-Baqarah/ 2: 164.

النَّاسَ يَنْفَعُ بِمَا الْبَحْرِ فِى تَجْرِىْ الَّتِىْ وَالْفُلْكِ وَالنَّهَارِ الَّيْلِ وَاخْتِلَافِ وَالْاَرْضِ السَّمٰوٰتِ خَلْقِ فِىْٓ اِنَّ
وَّتَصْرِيْفِ بَّةٍ دَآ كُلِّ مِنْ فِيْهَا وَبَثَّ مَوْتِهَا بَعْدَ الْاَرْضَ بِهٖ فَاَحْيَا مَّاۤءٍ مِنْ السَّمَاۤءِ مِنَ اللّٰهُ اَنْزَلَ وَمَاۤ
يَّعْقِلُوْنَ لِّقَوْمٍ لَاٰيٰتٍ وَالْاَرْضِ السَّمَاۤءِ بَيْنَ الْمُسَخَّرِ وَالسَّحَابِ الرِّيٰحِ

Terjemahnya:

Sesungguhnya pada penciptaan langit dan bumi, pergantian malam dan siang, kapal yang berlayar di laut dengan (muatan) yang bermanfaat bagi manusia, apa yang diturunkan Allah dari langit berupa air, lalu dengan itu dihidupkan-Nya bumi setelah mati (kering), dan Dia tebarkan di dalamnya bermacam-macam binatang, dan perkisaran angin dan awan yang dikendalikan antara langit dan bumi, (semua itu) sungguh, merupakan tanda-tanda (kebesaran Allah) bagi orang-orang yang mengerti (QS. Al-Baqarah/ 2: 164).

Surah Al-Baqarah verse 164 invites human beings to observe and reflect upon natural phenomena as signs of the greatness of Allah (SWT). In the context of this research, this message can be reflected upon through the effort to understand the characteristics of natural materials and industrial raw materials using a scientific approach. Testing the methylene blue index in a mixture of silica sand and clay serves as a means to determine how the material responds to the adsorption of dyes, as well as to understand how varying the clay composition influences the test results. This research also highlights the importance of precision in testing and evaluating raw materials, ensuring that their characteristics can be determined in a more quantifiable manner. The integration of this verse serves as a reflection on scientific values and the human responsibility to study and utilize natural resources; it does not, however, constitute the direct basis for the specific testing methodology employed.

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