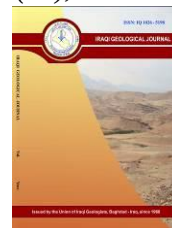




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Estimating Soil Plasticity Index Using the Methylene Blue Test for Soil Classification at Selected Sites in Hilla City, Babylon Governorate, Iraq

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Abstract

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Grain size distribution and Atterberg limits, with particular emphasis on the plasticity index as a key quantitative indicator, are vital for characterizing soil mechanical behavior and assessing its deformability and stability under various loads. These are the standard foundations upon which soil classification systems used in geotechnical applications are built. An operator-dependent procedure may influence the plasticity index determination. This study aims to evaluate the applicability of the methylene blue test as an alternative indicator for estimating the plasticity index in fine-grained soil. It is relatively uncommon compared to the Atterberg limits. Fifteen soil samples, to a depth of 30 cm, were collected from various locations in the center of Hilla City, Babylon Governorate. They analyzed grain size distribution, determined the Atterberg limits and specific gravity, and measured the methylene blue value according to ASTM and NF standards. Therefore, this study sought to investigate the relationship between the plasticity index and methylene blue, determine the nature of their correlation, and assess the possibility of employing this relationship in geotechnical evaluation. The soils studied, characterized by high contents of silty clay and clayey silt, were classified as CL, ML, and CL-ML. The linear regression analysis between methylene blue and plasticity index yielded a moderate correlation coefficient ($R^2 = 0.456$). The methylene blue value ranged from 8 to 30. These results indicate that the methylene blue test can provide a practical, preliminary estimate of soil plasticity index and support soil classification when rapid assessment is required. Still, it is not a direct substitute for Atterberg limits. It provides a practical initial tool for the rapid assessment and engineering classification of fine-grained soils.

Keywords: Soil classification; Geotechnical engineering; Methylene blue value; Atterberg limits; Plasticity index

1. Introduction

Soil classification can provide a basis for predicting engineering behavior and help in selecting appropriate construction and foundation solutions. Most commonly used geotechnical classification systems rely primarily on particle size distribution and plasticity properties. Soils with similar characteristics are classified into groups and subgroups based on their engineering behaviors. The soil classification in most systems developed for engineering applications relies on two essential criteria: plasticity and particle-size distribution. Despite the multitude of soil classification systems, no single system is universally applicable to all soil types and applications, given the considerable variability in their properties and engineering behavior (ASTM D 4318-10).

In the early 20th century, a refined method for describing the texture of fine soils with varying moisture content was proposed by Atterberg (a Swedish scientist). Soil behaves similarly to a solid at low moisture content. When the moisture content is very high, the soil may flow like a liquid. Thus, soil behavior can be arbitrarily divided into solid, semi-solid, soft, and liquid states, depending on moisture content (Fig. 1). The shrinkage limit is defined as the moisture content at which soil transitions from solid to semi-solid (Das, 2010). The moisture content at which the transition from semi-solid to plastic occurs is called the plastic limit, and the transition from plastic to liquid is called the liquid limit. These parameters are called Atterberg limits. Depending on Das (2010), the plasticity index (PI), determined from the Atterberg limits test, is defined as the difference between the liquid limit (LL) and the plasticity limit (PL) and is used as an indicator of soil plasticity or the range of plastic behavior.

When performing LL and PL tests by using the famous method (Casagrande method), human error that easily occurs when performing the examination can affect the subjectivity of PI values (Zhang et al., 2013). Fig. 2 shows the plasticity chart used in the Unified Soil Classification System (USCS) (ASTM D 2487-93, 1993).

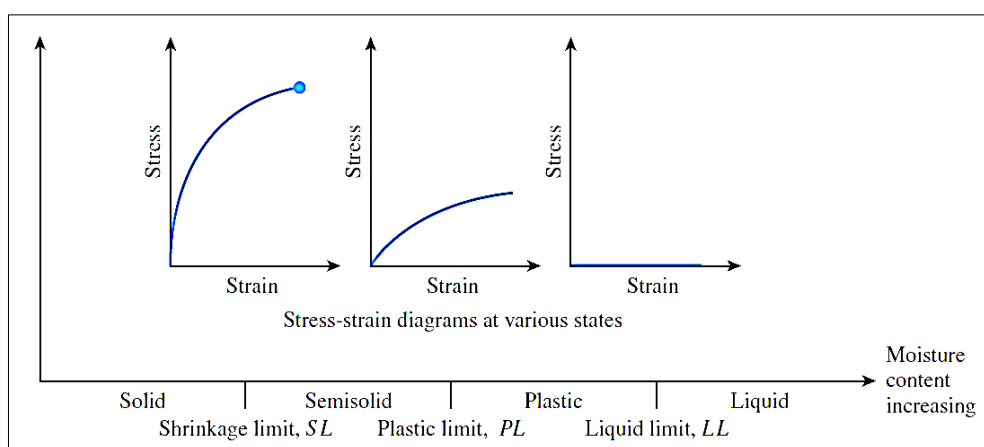


Fig. 1. Atterberg limits (Das, 2010)

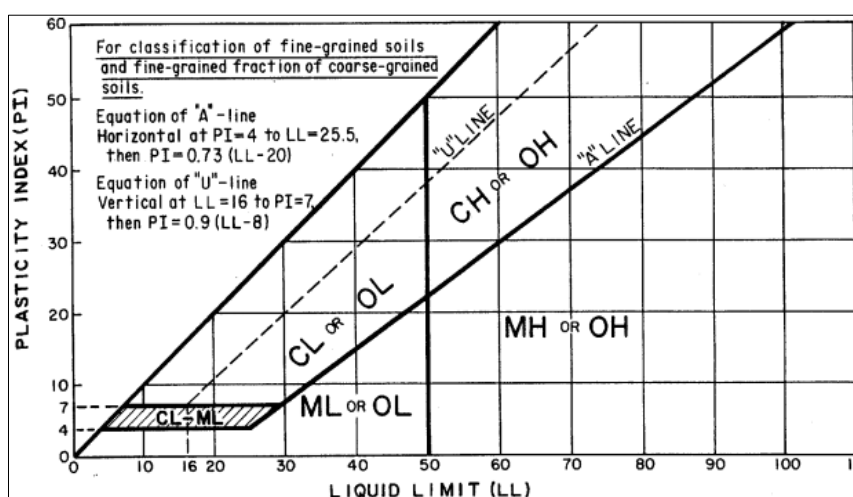


Fig. 2. The chart of plasticity for the USCS per ASTM D2487 (2011)

The falling cone method (another method) is widely used in Europe and Asia to determine the liquid limit, as shown in Fig. 3, where the LL represents the content of moisture at which a standard cone, with a vertex angle of 30° and a weight of 0.78 N (80 gf), penetrates a distance $d = 20$ mm in 5 seconds from its point of contact with the soil surface. Because a single test is insufficient to accurately

determine the liquid limit, four or more tests are usually carried out at different moisture contents to more reliably measure the falling cone penetration depth (d) (BS:1377, 1990).

To overcome this problem, other criteria were used that represent universal, fast, and reliable methods for comparison and determination of the PI (Seybold et al., 2008), one of which can be used to compare with the plasticity index is the methylene blue (MB) value. The methylene blue value may be obtained from the methylene blue test NF P 94-068, 1998, NF EN 933-9, 1999, and LCPC/ SETRA, 2003. The development of this test originated in France, where it was initially used to determine the suitability of aggregate materials for concrete production by measuring their clay content, a factor that influences their technical properties (Turkoz and Tosun, 2011). The spot measurement method is the most common approach to testing, and Chiappone et al. (2004) consider it a simplified calibration technique.

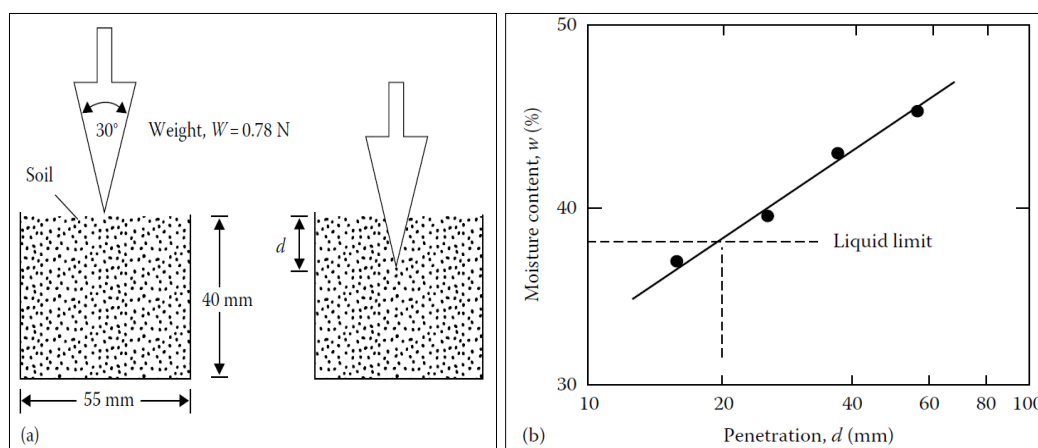


Fig. 3. a) Represented falling cone method. b) A binary graph represents moisture content against cone penetration to determine liquid limit (Das, 2019)

Seybold et al. (2008) studied the consistency limit, defined as the moisture content limit at which strength equals deformation, in the classification of soil moisture content. They emphasized its important role in classifying swelling potential and cohesion, as well as in describing soil properties such as compressibility, bearing capacity, and shear strength.

A series of studies was conducted by Chiappone et al. (2004) to assess the suitability of the MB test for identifying clay minerals, in accordance with the standard methods used in France and the United States. The results of these studies showed a discrepancy between the American and French methods. The test method specified in the American standard was suitable for fine, homogeneous materials; that is, it provides only information about the clay content. In contrast, the method specified in the French standard was suitable for heterogeneous materials, providing information representative of the material across all particle sizes.

Turkoz and Tosun (2011) predicted the swelling parameters (swelling ratio and swelling pressure) of natural clay soils in Harran City using the methylene blue test at a site in southeastern Anatolia. Thirty-three sites were selected, undisturbed samples were collected from them, and laboratory analyses were carried out using a recording device in an area with a high density of irrigation canals. In the developed models based on soil dry density and MBV, the R^2 values were 0.93 and 0.85 for the swelling ratio and swelling pressure, respectively.

Kuriakose et al. (2017) concluded that the natural water content ratio (NWCR) can be used instead of the liquidity index (L.I) because there is a significant linear relationship between the two. Otcu et al. (2017) indicated that the relationship between the PI and MBV, expressed in grams of dye per kg of the 0.2 mm fraction, is given by Equation 1.

$$PI = 1.1097 MBV^{0.7837} \quad (1)$$

This study primarily aims to explore the applicability of the MBV method for estimating the PI of fine-grained soils and to investigate the nature of the relationship between the plasticity index (PI) and methylene blue values (MBV), to obtain models or relationships that can be used to improve the accuracy of soil classification and interpret its geometric behavior.

1.1. Study Area

The study area is in Hilla City, approximately 100 km south of Baghdad in central Iraq. Its coordinates are 32°26' N to 32°32' N latitude and 44°23' E to 44°29' E longitude. Most of the land in the study area is generally flat, with occasional hilly areas (Manii et al., 2012). The Hilla River, which runs through most of the city and branches from the Euphrates River north of the governorate, in Al-Musayyib District, at Sadah City, contributes to the region's agricultural richness (Chabuk et al., 2017). Fig. 4 shows the locations of the samples and their corresponding coordinates.

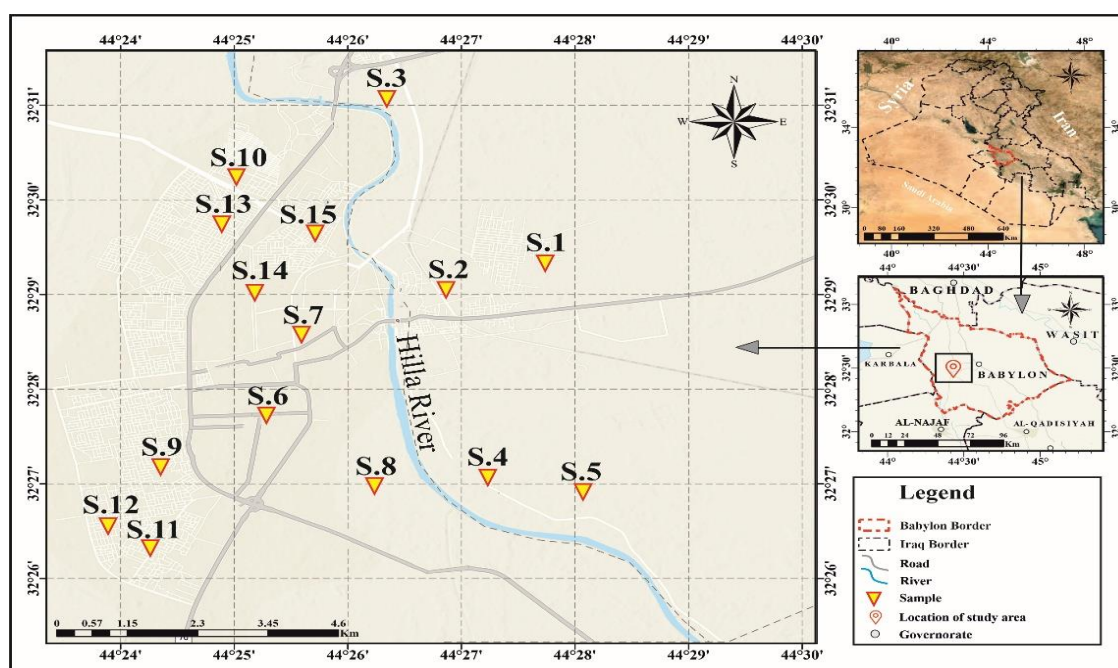


Fig. 4. Location map of samples in the study area

1.2. Geological Setting

The highest elevation point in Hilla City is approximately 28 m above sea level. This corresponds to the slope of the Mesopotamian plain, with a maximum elevation of 140 m a.s.l in the Al-Fathah Quarter in the north and a minimum elevation of approximately 1 m a.s.l along the far southeastern coast of the Gulf (Yacoub, 2011; Al-Rubaiee and Al-Owaidi, 2023). The soil in this region is alluvial, silty clayey, comparable to that throughout Mesopotamia, and results from deposition by the Euphrates and Tigris rivers. Geologically, Hilla City is situated within the unstable shelf sedimentary basin, as classified by the tectonic division of Iraq (Buday and Jassim, 1984; Al-Rubaiee and Al-Owaidi, 2024). It is located between the Zagros Mountains to the northeast and the stable Arabian Plateau to the south. This region is commonly referred to as the Mesopotamian Zone in the current geological classification of Iraq (Jassim and Goff, 2006) (Fig. 5).

2. Materials and Methods

The study area (Hilla City) was surveyed, and 15 soil samples were randomly collected and distributed throughout the area. The sample locations were selected based on engineering and environmental criteria, including selecting sites far from pollutants and construction waste. Various tests were conducted in this context on samples collected to a depth of 30 cm below the ground surface. Grain-size and dry- and wet analysis, in addition to tests of the Atterberg limits, were conducted to classify the soil samples according to the USCS. In the other stage, the percentages of gravel, sand, and fine materials (silt and clay) were determined. Tests were conducted according to the standards of the American Society for Testing and Materials: ASTM D-422, 2017; D2487-11, 2011; and D4318-10, 2010. Fig. 6 shows the device used to test the liquid and plastic limits with the Casagrande Method.

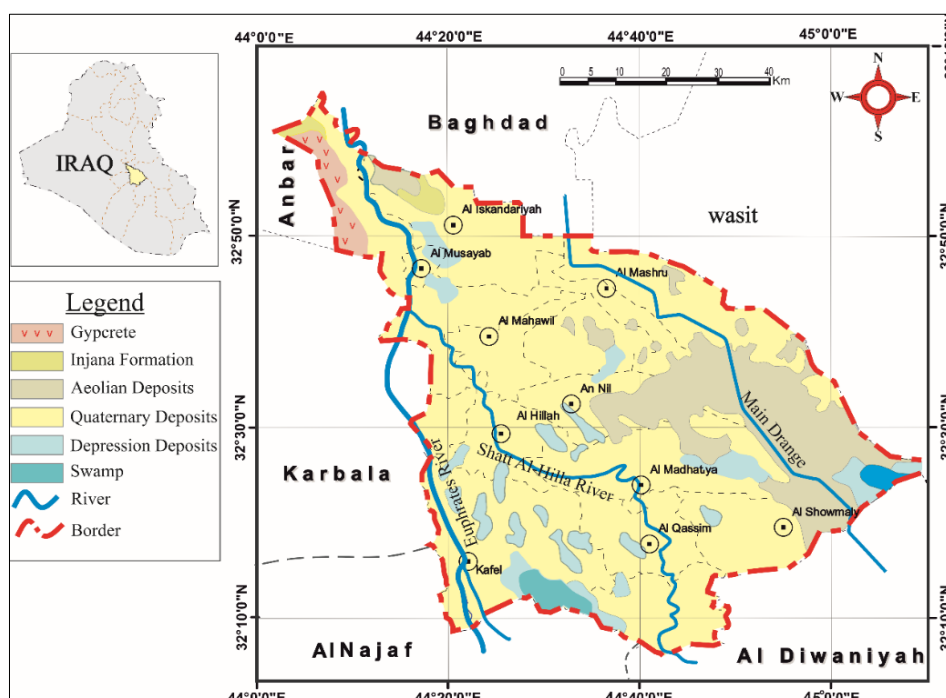


Fig. 5. Geological map of the Babylon Governorate (modified after Sissakian and Fouad, 2015)

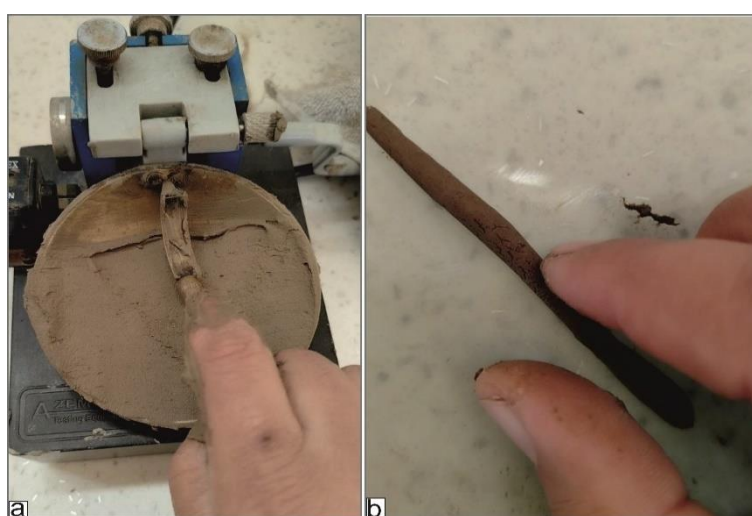


Fig. 6. a) Casagrande device, liquid limit test (LL), b) Rotating and rolling a soil sample mass on a polished glass panel to identify the plasticity limit, (after Das, 2010)

In this study, 15 fresh soil samples were collected from various sites across the governorate (Fig. 4), and their geographic coordinates are listed in Table 1. Laboratory experiments were conducted on each sample, and the methods used are briefly described below.

The relevant samples were classified as low-plasticity clays (CL and CL-ML) according to the USCS. The methylene blue test follows NF P 94-068 (1998) standards. The results were then analyzed to compare PI and MBV. The soil classification was based on the USCS, as specified in ASTM D2487-11 (2011).

2.1. Preparation of the Methylene Blue Solutions

Methylene blue tests were performed in accordance with the French standard NF P 94-068 (Afnor, 1993). To prepare a methylene blue solution, methylene blue powder is dissolved in a specific volume of distilled water. It was prepared by dissolving 10 ± 0.1 g of methylene blue powder in 1 liter of distilled water at room temperature, with continuous stirring for 1 hour to ensure complete dissolution and homogeneity. A soil suspension was prepared by passing 7.5 g of the sample through a No. 40 (0.425 mm) sieve and mixing it with 50 ml of distilled water in a glass beaker. If using a 30 g sample, 200 ml of distilled water was added. The mixture was stirred with a mechanical mixer at 700 rpm for 5 minutes to ensure a homogeneous suspension prior to testing (Çokça, 1991).

Table 1. Coordinates of sites in the study area

Sample No.	Name of Quarter or area	Latitude	Longitude
1	Al-Bakrli-1	32°29'38.02"N	44°27'33.35"E
2	Al-Khosrawiya	32°29'19.42"N	44°26'37.27"E
3	Algiers	32°30'55.76"N	44°26'26.40"E
4	Kretaa	32°28'25.45"N	44°26'43.17"E
5	Al-Thila	32°27'58.32"N	44°26'33.07"E
6	Al-Askan	32°28'0.33"N	44°24'48.91"E
7	Al-Jameia	32°28'22.23"N	44°25'3.60"E
8	Nader-1	32°27'3.52"N	44°25'43.15"E
9	Mohandessin-1	32°27'1.15"N	44°24'12.64"E
10	Al-Thawra	32°30'16.26"N	44°25'5.03"E
11	30th Street	32°26'36.27"N	44°24'28.06"E
12	Al-Askari Quarter	32°26'22.71"N	44°23'57.63"E
13	Al-Imam Quarter	32°29'52.65"N	44°24'56.89"E
14	Al-Karamah-1	32°29'14.83"N	44°25'10.28"E
15	Al-Tayyara Quarter	32°29'43.50"N	44°25'33.32"E

2.2. Methylene Blue Analysis

The methylene blue test, developed in France, was used to detect the clay content in aggregate materials and to determine their suitability for producing concrete. Methylene blue powder acts as a cationic dye when dissolved in water and has the chemical formula $C_{16}H_{18}N_3SCl$. During mixing of the methylene solution with the soil solution, chloride ions in the methylene structure will replace cations in clay minerals and then be adsorbed onto the clay mineral surfaces. The amount of methylene blue solution absorbed varies with the proportions and types of clay minerals in the sample, as well as with the cation exchange capacity and specific surface area. These factors combined are key determinants of adsorption behavior in soil.

Due to its ease of application and the lack of need for special equipment, this test (the methylene blue test) is a common method for determining the mineral content of clay. Therefore, this test is

considered an economical, simple, and reliable method, particularly in the early stages of research (Verhoef, 1992).

2.2.1. Methods for testing Methylene blue

Generally, to obtain reliable results, two testing methods are used: turbidimetric and spot measurement. The turbidity measurement method relies on measuring the absorption of methylene blue dye on clay surfaces. Unabsorbed dye in solution causes turbidity proportional to the available surface area (Lamar, 1954). Whereas the spot measurement, which was used in this study, applied according to ASTM C1777, 2015, involves suspending 25 grams of soil (particle size $<75\ \mu\text{m}$) in water, titrating it with methylene blue solution until the soil is saturated (blue spot test endpoint), filtering the solution, diluting the filtrate, and measuring the absorption at 670 nm using a colorimeter to calculate the methylene blue value (MBV = mg/g soil). A higher MBV value ($>2.0\ \text{mg/g}$) indicates the presence of swollen clay, which causes problems and leads to deterioration in concrete performance through increased water demand and shrinkage.

Spot measurement is a simplified titration technique in which a specific volume of a methylene blue solution at a given concentration is added to a suspension of fine soil. The total volume of methylene blue solution absorbed is used to calculate the MBV. This method is widely used in many engineering applications (Taylor, 1985). The properties of the indicator (such as the liquid limit and plasticity index) combined with the MBV enable important assessments, particularly in preliminary site engineering studies. The cation exchange capacity (Taylor, 1985) and specific surface area (Chiappone et al., 2004) can be assessed using the methylene blue test. This test determines the soil's ability to absorb ions by measuring the amount of methylene blue required to completely cover the clay particles.

This method uses titration, which involves a chemical reaction between free cations of methylene blue, produced from the methylene blue solution, and cations on clay particles. Clay particles with a larger specific surface area and a higher negative charge exhibit the greatest cation exchange capacity. Adsorption capacity increases with increasing specific surface area and electrical charge of the clay particles (Chiappone et al., 2004). The French standard NF P 94-068 (1998) (Afnor, 1993) and the American standard C837-99 (ASTM, 1999) have conducted comparative studies on the feasibility and evaluation of the methylene blue test for identifying clay minerals.

Afnor (1993) standard recommends a standardized soil sampling analysis based on clay content. For clay or very clay soils, 30–60 grams are used; for less-clay soils, 60–120 grams. Soil samples were taken in predetermined proportions and suspended in 500 ml of distilled water, stirring to ensure homogeneity. Then, 5 ml of 10 g/l methylene blue solution was added to the soil suspension. After 1 minute of stirring, a drop of the mixture was placed on filter paper to observe the adsorption spot and determine the endpoint. The test concludes with the formation of a blue dye halo around the spot, which must remain stable for 5 minutes. The ASTM standard, based on the same procedure as the AFNOR standard (Afnor, 1993), uses a 2 gm soil sample and performs the analysis under acidic conditions (pH 2.5–3.8). The ASTM standard test is suitable for homogeneous, fine-grained soils that only assess clay content, whereas the Afnor (1993) method provides results for the entire material; therefore, it is more suitable for heterogeneous samples (Chiappone et al., 2004).

2.2.2. Procedure, calculations, and analysis

The mixture was prepared by adding increasing amounts of methylene blue solution to the soil suspension. After adding 5 ml of a 10 g/l methylene blue solution, the mixture was stirred at 400 rpm using a mixer and maintained at a steady speed throughout the test to ensure homogeneity. After 1 minute of mixing, the mixture was drawn off with a glass pipette, and a drop was placed on a filter paper. The filter paper was placed on a glass beaker so that its wetted surface did not come into contact with

any solid or liquid. Typically, when the first drop was placed on the filter paper, a dark blue spot surrounded by a colorless, moist halo appeared, indicating a negative result and reflecting the clay particles' lack of saturation with the methylene blue solution at this stage. Five ml was added to the soil solution until a halo appeared around the dark blue spot on the filter paper.

The mixture was checked every minute for up to 5 minutes to assess the stability of the light-blue halo. At the end of the test, if the light blue halo around the dark blue spot was surrounded by a region of clear water (indicating a positive result), this indicated that excess methylene blue was no longer absorbed by the clay mineral and remained suspended. If the light blue ring around the spots disappeared within one minute, the amount of methylene blue solution added was reduced to 2 ml, and the same procedure was followed.

Fig. 7 shows a flow diagram of the methylene blue staining test. The methylene blue value (MBV) is typically described in grams.

The amount of methylene blue absorbed by 100 g of sample material is usually expressed in grams per 100 g. The MBV value for the sample was calculated by using Equation 2.

$$MBV \left(\frac{g}{100g} \right) = \frac{V_{cc}(mL)}{f'(g)} \quad (2)$$

Where V_{cc} = methylene blue solution volume that was added to the soil solution (ml), f' = weight of the dry sample (g).

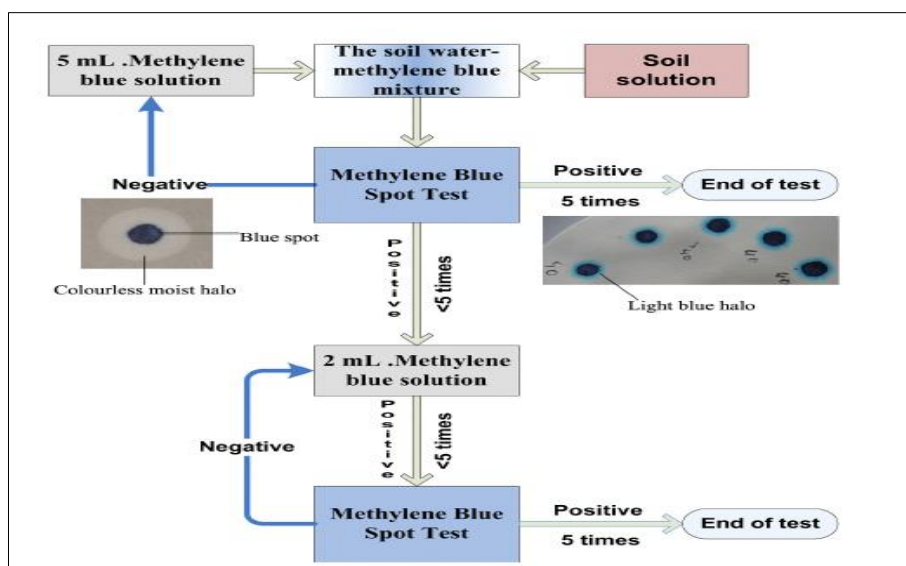


Fig. 7. Flowchart illustrating the steps of the methylene blue spot test (after Türköz and Tosun, 2011)

3. Results and Discussion

3.1. Moisture Content

The moisture content values of the selected soils in Hilla City ranged from 5 to 31%, as shown in Table 2, indicating that some soils were moister than others owing to the relatively high soil moisture resulting from the proximity of groundwater levels in the study area.

3.2. Specific Gravity

This test was conducted on natural soil samples in accordance with ASTM D-854 (2010). A glass pycnometer was used in the procedure, in which a dry soil sample weighing no more than 100 g was placed. The remaining steps of the method were followed to determine the specific gravity (Gs) of each

soil sample. The specific gravity values ranged between 2.71 and 2.75, as listed in Table 2. This indicates that the predominant soils at the study sites were fine-grained silty clay sand or clayey silty sand soils.

Table 2. Results of grain size analysis test, MBV, and plasticity index for selected samples

No.	Area Name	Moisture Content %	Specific Gravity	Atterberg Limits%			Grain size analyses			MBV
				L.L %	P.L %	P.I %	Silt%	Clay%	Gravel & Sand%	
1	Al-Bakrli-1	10	2.72	32	22	10	49	37	14	20
2	Al-Khosrawiya	10	2.72	25	12	13	43	45	12	30
3	Algiers	5	2.72	27	22	5	45	44	11	11
4	Kreta'a	7	2.72	23	19	4	41	46	13	10
5	Al-Thila	18	2.71	24	12	12	36	48	12	19
6	Al-Askan	9	2.75	24	13	11	39	45	16	18
7	Al-Gamaeah	17	2.74	23	20	3	44	21	35	8
8	Nader-1	2	2.75	29	19	10	39	43	18	20
9	Mohandessin-1	13	2.74	26	19	7	44	40	16	12
10	Al-Thawra	14	2.75	27	18	9	42	47	11	15
11	30 th Street	23	2.74	22	15	7	46	37	17	10
12	Al-Askari Quarter	12	2.74	28	18	10	36	48	16	18
13	Al-Imam Quarter	15	2.74	24	15	9	40	45	15	25
14	Al-Karamah-1	13	2.74	29	19	10	36	44	20	22
15	Al-Tayyara Quarter	31	2.73	34	28	6	36	30	34	13

3.3. Atterberg Limits Test

The operator's technique strongly influences the results of the Atterberg limit. For instance, determining the number of blows required to close the groove during the liquid limit test is subject to individual judgment. Similarly, the pressure exerted by the technician's palm during soil rolling affected the measured plastic limit. In contrast, the methylene blue test using the spot method is less operator-dependent. This is because its procedure is more straightforward, making the test easier to perform and yielding consistent results.

3.4. Grain Size Analyses

The grain size distribution was determined using two main procedures: sieve and hydrometer analysis. A 200 g soil sample was prepared and washed over a No. 200 sieve (0.075 mm) to separate the gravel, sand, silt, and clay fractions. All testing procedures were conducted in accordance with ASTM D-422 (2017), the standard method for particle size analysis of soil. Table 2 presents the results of the grain-size analysis for samples from the study area.

3.5. Correlation Between Plasticity Index and Methylene Blue Values

The relationship shown in the equation was derived from the blue methylene and plasticity index values measured using the Casagrande device test. The findings of this study indicate that for MBV ranging from 8 to 30, the relationship can be represented by a linear equation with a determination coefficient of $R^2 = 0.4559$, as shown in Fig. 8. Table 3 shows the plasticity index values obtained from the blue methylene values after using the following experimental equation:

$$PI = 0.4975 MBV - 0.7921 \quad (3)$$

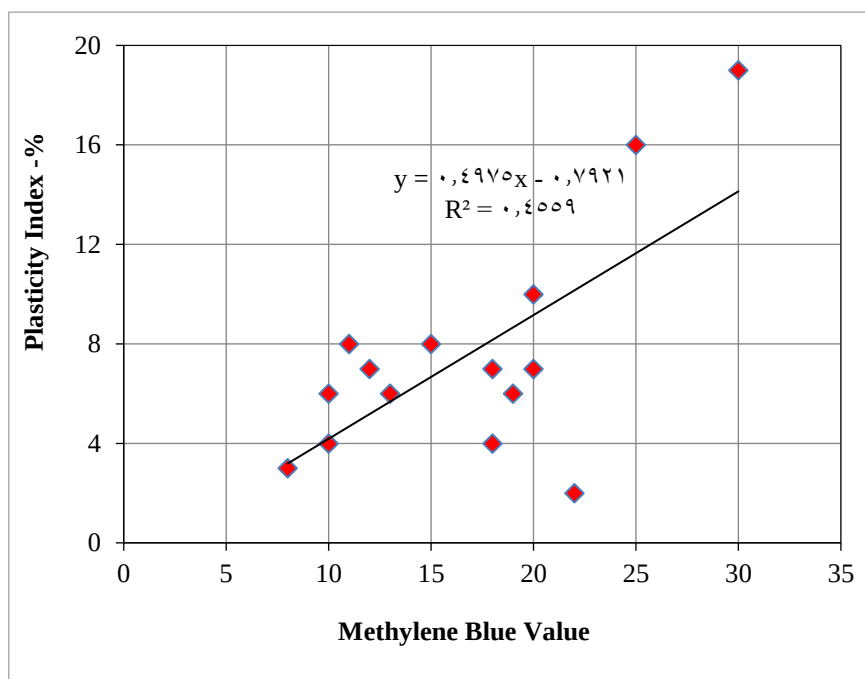


Fig. 8. Curve of plasticity index and methylene blue values

Table 3. Comparison and correlation of Plasticity Index Determined from Atterberg Limits and Methylene Blue Value (MBV) Test Results

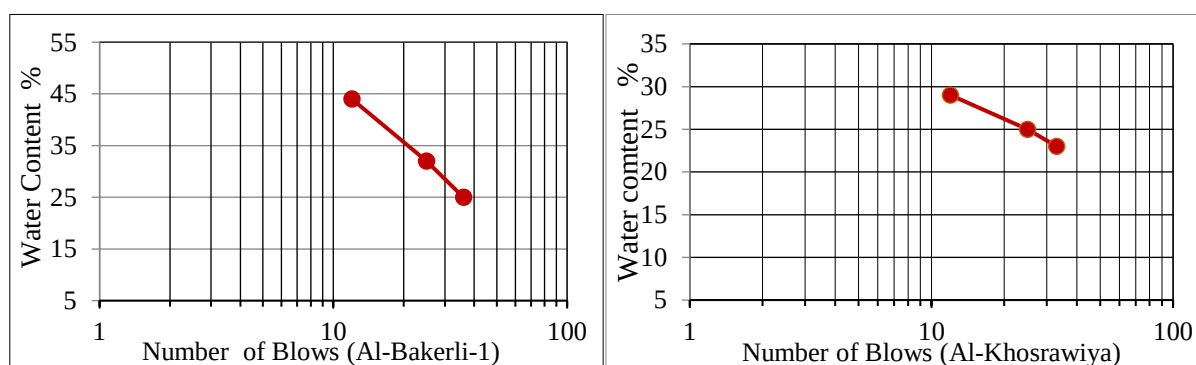
No.	PI = L.L – P.L From Atterberg limits	MBV	PI = 0.4975 MBV – 0.7921 From Correlation with MBV
1	10	20	9
2	13	30	14
3	5	11	5
4	4	10	4
5	12	19	9
6	11	18	8
7	3	8	3
8	10	20	9
9	7	12	5
10	9	15	8
11	7	10	4
12	10	18	8
13	9	25	12
14	10	22	9
15	6	13	6

3.6. Soil Classification

Based on the blue methylene values shown in Table 3 and Fig. 8 compared to the Atterberg Limits Test (Fig. 9), the soil samples at the study sites were classified according to the Unified Soil Classification System (USCS) (ASTM D 2487-93, 1993). The predominant soils were classified as follows: ML (silty soil low plasticity), CL (clay soil low plasticity), and CL-ML (Table 4).

Table 4. Soil classification based on USCS

Sample No.	Group Name	Group Symbol
	Soil classification USCS	
1	Silty clay sand	ML
2	Clayey silty sand	CL
3	Silty clay sand	CL-ML
4	Silty clay sand	CL-ML
5	Clayey silty sand	CL
6	Clayey silty sand	CL
7	Silty clay sand	ML
8	Clayey silty sand	CL
9	Silty clay sand	CL-ML
10	Clayey silty sand	CL
11	Silty clay sand	ML
12	Clayey silty sand	CL
13	Clayey silty sand	CL
14	Clayey silty sand	CL
15	Silty clay sand	ML

**Fig. 9.** Atterberg Limits Test of the study area

4. Conclusions

The Plasticity Index (PI) is a key soil parameter used for classification under the Unified Soil Classification System (USCS). It can be estimated by correlating it with the MBV obtained from the methylene blue test. Compared to the Atterberg limit test, the methylene blue procedure is simpler and more practical.

The results of this study indicate that within the MBV range of 8 - 30, the relationship between MBV and the plasticity index is linear, with a coefficient of determination of $R^2 = 0.4559$. This result indicates the potential to use the estimated PI values derived from this relationship, when combined with grain-size analysis results, to support soil classification under the USCS system with acceptable efficiency.

Author contributions: CRediT

Conceptualization: Dr. Abdul-Kareem H. Al-Rubaie; Methodology: All authors; Investigation: All authors; Data Curation: All authors; Formal Analysis: All authors; Writing – Original Draft: All authors; Writing – Review & Editing: All authors; Visualization: All authors; Supervision: the authors together

Declaration of Competing Interest

The authors declare that they have no competing financial interests or personal relationships that could influence the work reported in this study.

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Data Availability

All data generated or analyzed during this study are included in this published article and/or its supplementary materials

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