

RELIABILITY OF METHYLENE BLUE EXCHANGE METHODS FOR ESTIMATION OF CATION-EXCHANGE CAPACITY OF DIFFERENT EGYPTIAN SOILS AND PURE CLAY MINERALS

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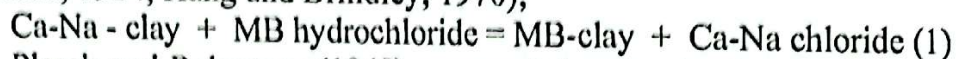
This study was carried out to certify methylene blue (MB) exchange method as a rapid one for determination of cation-exchange capacity CEC of different Egyptian soil locations having a wide range of variation in soil physical and chemical properties as soil texture, structure, CaCO_3 , pH, O.M, and CEC as well as three pure clay minerals, namely: Na-montmorillonite (Wyoming, U.S.A); Palygorskite (Georgia, U.S.A) and Kaolinite (Scalby, England). Four methods were tested to estimate CEC of such soils and these 3 pure clay minerals. These methods were conventional Na-NH_4 exchange; MB-acid, MB-tetra sodium pyrophosphate (TSPP) and MB-water filter paper spot test titration methods. Soil samples were analyzed under suitable conditions of concentration, equilibration period and flocculation of MB solutions. The squared regression coefficient (R^2) of MB-TSPP vs. Na-NH_4 exchange methods for soil samples was 0.98. The R^2 of MB-water vs. Na-NH_4 exchange methods for pure clay mineral samples was 1.0. The MB test is a simple, rapid and economical method to determine CEC. The CEC values measured by MB-TSPP were closed to the calculated values for different Egyptian soils. But the values determined by MB-water were closed to the calculated values for pure clay minerals. The MB-TSPP and MB-water exchange methods may be suitable for the field-testing of soils and pure clay minerals, respectively.

Keywords: cation exchange capacity, clay minerals, methylene blue methods.

The ability of a soil to supply nutrients for plant growth is an important factor of soil fertility. The concept of soil fertility includes not only the quantity of nutrients a soil contains, but how well nutrients are protected from leaching, how available the nutrients are, and how easily roots can grow. The amount of storage capacity of a soil for nutrients is, among others,

expressed by the cation exchange capacity (CEC) and is affected by some soil physical properties, such as texture and consistence. A number of other differences among soils are related to CEC. For instance, the amount of lime, gypsum, fertilization practices and how much herbicide should be spread on the soil, often depend on the CEC (Plaster, 1992).

Cation-Exchange capacity is of fundamental and practical importance in research involving soils and clay minerals. Methylene blue (MB) is widely used in industry. Jones (1964) introduced MB in a test to determine bentonite in drilling fluids. This method, widely known as the "MB test", was added to the American Petroleum Inst. RB13B standard procedure for testing drilling fluids in 1966. This test is based on adsorption of MB dye from solution by a clay surface. Methylene blue consists of an organic cation and an anion. The cross-sectional area of the molecule is 1.35 nm^2 (Kipling and Wilson, 1960). When the dye is dissolved in water MB exists mainly as monomers at low concentrations (below $7 \times 10^{-6} \text{ M}$) and in a monomer-dimer equilibrium at higher concentrations. Methylene blue replaces natural cations irreversibly, unlike reversible exchange of inorganic cations as follows: (Bascomb, 1964, Hang and Brindley, 1970);



Plesch and Robertson (1948) proposed that the dye is adsorbed in two ways: irreversible exchange of an amount equivalent to CEC and reversible physical adsorption, which can be described by a Freundlich isotherm. Their proposal is consistent with that of Hills and Pettifier (1985) Taylor (1985). Soon (1988) and Rytwo *et al.* (1991) developed the MB-adsorption method using a spectrophotometer at a 665-nm wavelength to measure the CEC and surface area of clays, soils and porous materials.

The objective of this work was to testify this MB method as a rapid one for the determination of CEC of different Egyptian soils having a wide range of variations in soil physical and chemical properties as soil texture, structure, CaCO_3 , pH, O.M. and CEC.

MATERIALS AND METHODS

The area under investigation is bordered east by the El-Tina plain, west by the El-Sharkiya Governorate, north by the Alexandria and south by the Ismailia canal as shown in fig. (1). Twenty-one representative soil samples collected from nine soil profiles differing in their texture where ranged from loamy sand to clay and were air-dried, ground and passed through a 2 mm sieve. Some physical and chemical properties of the soils under investigation are set up in table (1). Determination of properties was carried out as described by Jackson (1967), Richards (1954) and Klute (1986). Pure clay minerals were Na-montmorillonite (Wyoming, U.S.A.), Palygorskite (Georgia, U.S.A) and Kaolinite (Scalby, England).

TABLE (1). Physical and chemical properties of the investigated soils.

Location & Profile No.	Depth, cm	Sand		Silt		Clay	Textural class	O.M	CaCO ₃	pH	EC	Cations (me ^l _l ⁻¹)				Anions (me ^l _l ⁻¹)			
		%	%	%	%							Na ⁺	K ⁺	Ca ⁺⁺	Mg ⁺⁺	CO ₃ ⁼	HCO ₃ ⁻	Cl ⁻	SO ₄ ⁼
Burg El-Arab 1	0-25	55.9	16.7	27.4			SCL	0.56	28.89	8.10	8.00	64.6	3.50	13.2	12.3	0.00	4.00	85.0	4.60
	25-60	54.1	20.0	25.9			SCL	0.40	39.99	8.30	5.60	42.6	1.60	10.5	9.60	0.00	7.00	55.0	2.30
	60-120	62.1	24.4	13.5			SCL	0.37	52.22	8.50	2.20	15.8	0.60	3.90	2.70	0.00	5.00	9.60	8.40
	120-150	61.6	18.3	20.1			SCL	0.41	61.11	7.80	26.40	116.8	3.70	42.4	64.2	0.00	3.00	210.0	14.1
Wadi El-Tumilat 2	0-7	39.6	10.9	49.5			C	0.90	12.00	8.40	1.70	12.6	0.50	1.00	1.00	1.00	2.50	4.30	7.31
	7-25	37.4	3.60	59.0			C	1.14	13.29	9.60	2.50	18.3	0.50	0.50	0.50	5.00	2.00	4.80	7.98
Wadi El-Tumilat 3	0-10	48.0	4.10	47.9			SC	0.78	10.24	8.50	2.40	15.0	0.40	3.50	3.50	0.00	2.50	13.6	6.26
	10-30	59.2	0.30	40.5			SC	1.11	8.00	8.50	0.60	3.50	0.30	3.00	1.00	0.00	2.50	0.90	4.37
Ganakiis 4	0-25	75.6	13.0	11.4			SL	1.38	14.40	8.00	2.05	2.07	1.02	8.50	8.00	0.00	5.00	1.50	14.0
Abou-El-Matamir 5	0-30	80.3	9.20	10.5			SL	1.18	7.23	8.04	2.60	9.83	1.00	10.0	3.50	0.00	5.00	2.00	19.0
El-Ghar 6	0-30	20.6	20.3	55.6			C	1.50	2.10	7.30	0.28	1.64	0.11	1.01	0.23	0.00	1.82	0.70	0.48
	30-60	15.5	23.3	56.0			C	1.10	1.90	7.40	0.27	2.04	0.12	0.63	0.11	0.00	1.97	0.73	0.20
	60-90	14.8	22.5	62.0			C	0.90	1.70	7.30	0.28	2.39	0.10	0.40	0.31	0.00	1.94	0.69	0.56
El-Zankalon 7	0-30	8.5	10.5	78.4			C	1.80	1.90	7.00	0.34	2.48	0.06	0.40	0.66	0.00	1.50	0.70	1.40
	30-60	8.5	28.2	58.4			C	0.90	2.10	7.30	0.27	2.25	0.08	0.30	0.37	0.00	1.75	0.53	0.72
	60-90	9.4	30.3	54.9			C	0.70	1.70	7.40	0.25	2.02	0.09	0.59	0.10	0.00	1.75	0.67	0.38
Herret Razana 8	0-30	59.1	16.1	24.8			SCL	1.90	2.30	7.00	0.16	0.67	0.11	1.02	0.11	0.00	0.31	0.88	0.72
	30-60	57.8	16.7	25.5			SCL	0.90	2.10	7.20	0.15	1.02	0.07	0.40	0.30	0.00	0.44	0.69	0.66
	60-90	57.5	16.9	25.6			SCL	0.80	1.80	7.40	0.23	1.75	0.13	0.47	0.15	0.00	0.35	0.81	1.34
El-Tina plain 9	0-35	17.7	34.0	48.3			C	0.29	1.64	7.40	97.00	459.5	12.0	174.0	464.5	0.00	2.40	897.7	209.9
	35-60	85.9	4.00	10.1			LS	0.17	1.29	7.30	44.30	374.40	5.60	94.5	115.5	0.00	2.40	398.4	189.2
SCL= sandy clay loam												C= clay		SC: sandy clay		SL= sandy loam		LS= loamy sand	

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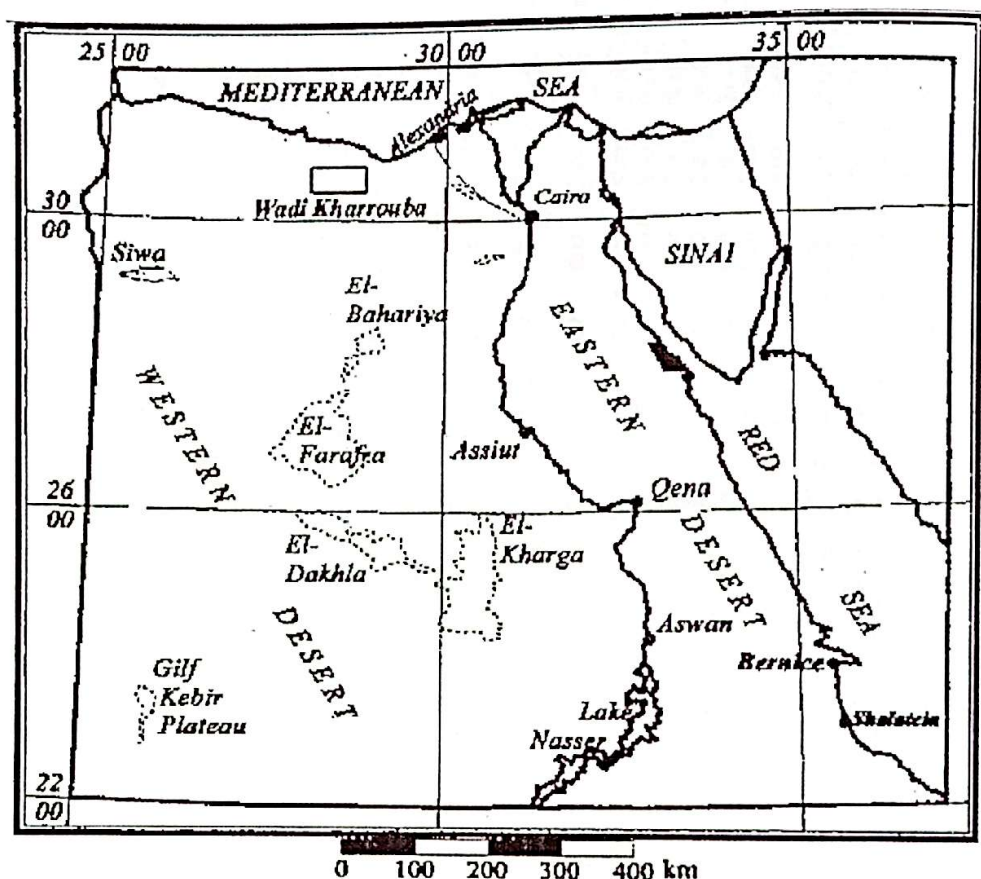


Fig. (1). Location map of the studied soil profiles.

All CEC analyses were done in duplicate. Four methods were used for comparison: Traditional Na-NH₄ exchange, MB-acid, MB-TSP (tetra sodium pyrophosphate) and MB-water filter paper spot tests. They could be described as follows: For traditional Na-NH₄ exchange method, determination of CEC was done in centrifuge tubes using oven-dry soil samples (6.0 g). Each sample was saturated with Na⁺ by washing with 132 ml of 1.0 N NaOAc solution buffered at pH 8.2. The electrolyte was washed from the samples with ethanol, 95 percent (99ml) and finally, exchangeable Na was displaced with (100 ml) of (1.0 N) NH₄OAc solution buffered at pH= 7. Displaced Na⁺ was determined flame photometrically as described by Rhoades (1982). For the MB-acid filter paper spot-test titration (American petroleum Inst., 1966), a sample (0.57 g) was weighed into an Erlenmeyer flask; H₂O₂ (300g l⁻¹, 10 ml) and H₂SO₄ (2.5 M, 0.5 ml) were added and the mixture was boiled gently for 5 min, then diluted to ≈ 50 ml with distilled water. The dye used was methylene blue hydrochloride (MB) (C₁₆H₁₈N₃SCl. 3H₂O). The MB solution (0.01 M) was added with a micropipette to the flask. After each added aliquot (0.5 ml), the contents of the flask were swirled to about half a minute. A drop of liquid was taken from this flask,

and the suspension was placed on a filter or test paper. The end point of the titration was marked by the appearance of the dye as a faint blue ring or halo surrounding the dyed solids. To express the CEC of soils or clays in centimole charge of saturating ion per kilogram the following equation was used:

$$\text{CEC} = \frac{\text{volume (milliliters) of MB-solution consumed}}{\text{weight of sample}} = \frac{\text{volume (milliliters) of MB solution consumed} \times 1.75}{\text{weight of sample}} \quad (2)$$

The MB-TSPP titration for filter paper spot test method is similar to the MB-acid procedure and is an empirical method. A solution of tetra sodium pyrophosphate (TSPP) (20 g l^{-1}) replaced the H_2SO_4 in MB-acid method. Soil or clay mineral sample (0.5 g) was transferred into an Erlenmeyer flask (250 ml) containing TSPP solution (20 g l^{-1} , 50 ml , $\text{pH} \approx 10$). The mixture was boiled on a hot plate gently, and distilled water was added as required to keep the volume near 50 ml . The flask was then removed from the heat and cooled to room temperature. The samples were titrated with MB solution (0.01 M); MB solution (1 ml) is equivalent to CEC $2 \text{ cmol}_c \text{ kg}^{-1}$ by the weight of oven-dried soil or clay mineral. For the MB-water titration method, distilled water was simply added to a sample suspension, followed by titration with MB solution and a filter paper spot test. Soil or clay mineral was not treated with H_2O_2 , H_2SO_4 or sodium pyrophosphate solution. The sample mass and CEC calculation were the same as for the MB-acid method.

RESULTS AND DISCUSSION

The determination of the cation exchange capacity is one of the most important analyses of soil physicochemical properties. The pH, organic matter, and CaCO_3 content of soils in table (1), can indicate a wide range of variations. The soil texture ranged from clay to sandy loam. Most soils were neutral and slightly or moderately alkaline. The NH_4OAC method was used as a reference one because it is used most commonly to determine CEC and exchangeable cations. Gillman *et al.* (1983) reported that a modified version of this procedure was precise (coefficient of variance = 1.3) and suitable as a standard method.

The MB-TSPP filter paper spot-test titration method gave CEC results of soils and pure clay minerals that significantly correlated with the Na- NH_4 exchange method (Table 2 and Figs. 2 to 7). The regression analysis indicated that CEC tends to be underestimated when measured by the MB-acid and MB-water titration methods. Such underestimation of CEC values is possibly caused by extensive aggregation, formation of H^+ or Al^{3+} clay with H_2SO_4 treatment or inhibition of MB sorption (Coleman and Harward, 1953; Van Olphen and Fripiat, 1979; Wang and Wang, 1987). Most CEC values of the soil samples measured

with Na-NH₄ exchange method were greater than those tested by MB-acid and MB-water methods, but lower than those by MB-TSPP titration method.

However, the CEC values of each of montmorillonite, palygorskite and kaolinite did not differ significantly by any of the studied methods as shown in table (2). Yet, the values obtained by MB-TSPP method tended to be greater than those by Na-NH₄ exchange, MB-acid or MB-water method. This finding could be explained by the smaller cation-exchange sites under the conditions of MB-acid and MB-water methods due to the lower pH of the suspension as compared with the conditions of MB-TSPP method where the pH is higher (pH ~ 10). The exchange sites, under such higher pH values, are completely saturated with MB giving greater CEC values. Wang and Wang (1987) reported that the CEC determination depends on pH, resulting in increasing CEC values under alkaline conditions due to formation of aluminates or silicates or metasilicates. While, under strongly acidic conditions, a decrease in CEC values can occur due to formation of H⁺ or Al³⁺ -exchange clays.

TABLE (2). Cation-exchange capacity (CEC) of soils and pure clays determined by Na-NH₄ exchange, MB-acid, MB-TSPP and MB-water filter paper spot test titration methods.

MB water after paper spot test titration methods.							
Location&Profile No.	SampleNo.	Depth,cm	CEC determination methods				
			Na-NH ₄	MB-acid	MB-TSPP	MB-water	
me100 g ⁻¹ soil							
Burg El-Arab	1	1	0-25	13.10	13.05	15.11	13.15
		2	25-60	12.80	12.85	13.33	12.65
		3	60-120	9.80	9.60	11.21	9.45
		4	120-150	11.10	10.89	14.05	10.75
Wadi El-Tumilat	2	5	0-7	31.50	30.15	29.99	31.00
		6	7-25	38.50	35.95	42.15	37.05
Wadi El-Tumilat	3	7	0-10	31.53	28.11	35.14	30.19
		8	10-30	31.50	26.15	36.00	28.95
Ganaklis	4	9	0-25	13.05	10.59	15.00	12.14
Abou El-Matamir	5	10	0-30	11.40	9.76	13.35	10.34
El-Ghar	6	11	0-30	34.60	30.15	41.03	31.88
		12	30-60	33.40	35.14	37.11	33.95
		13	60-90	32.50	29.80	34.75	30.14
El-Zankalon	7	14	0-30	32.50	30.45	34.14	31.00
		15	30-60	31.50	29.99	33.75	30.13
		16	60-90	13.25	11.85	15.91	12.05
Herret Razana	8	17	0-30	24.11	22.25	27.78	23.10
		18	30-60	24.32	21.15	28.11	23.18
		19	60-90	23.13	22.10	25.37	21.05
El-Tina Plain	9	20	0-35	32.55	30.15	36.15	39.81
		21	35-60	9.89	7.19	11.90	8.01
			F-test	L.S.D.			
Method (M)			..	0.298			
Depth (D)			..	0.683			
M × D			..	1.366			
Pure clay minerals	Na-Montmorillonite		75.14	69.63	74.32	70.85	
	Palygorskite		17.22	16.89	25.11	15.99	
	Kaolinite		4.05	3.90	4.19	4.00	
			F-test	L.S.D.			
			n.s	-			

** P<0.01

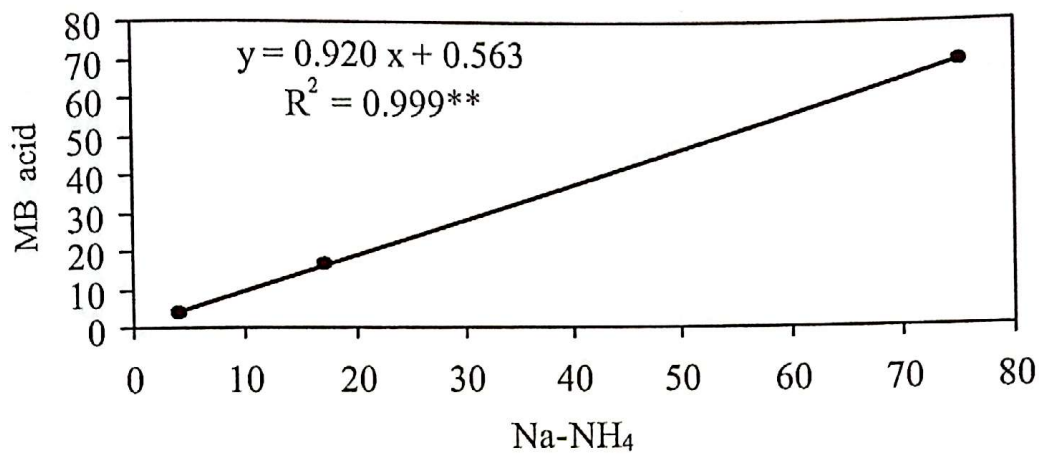


Fig. (2). Relationship between Na-NH₄ and MB-acid methods for determination of CEC (me100g⁻¹) in pure clay minerals.

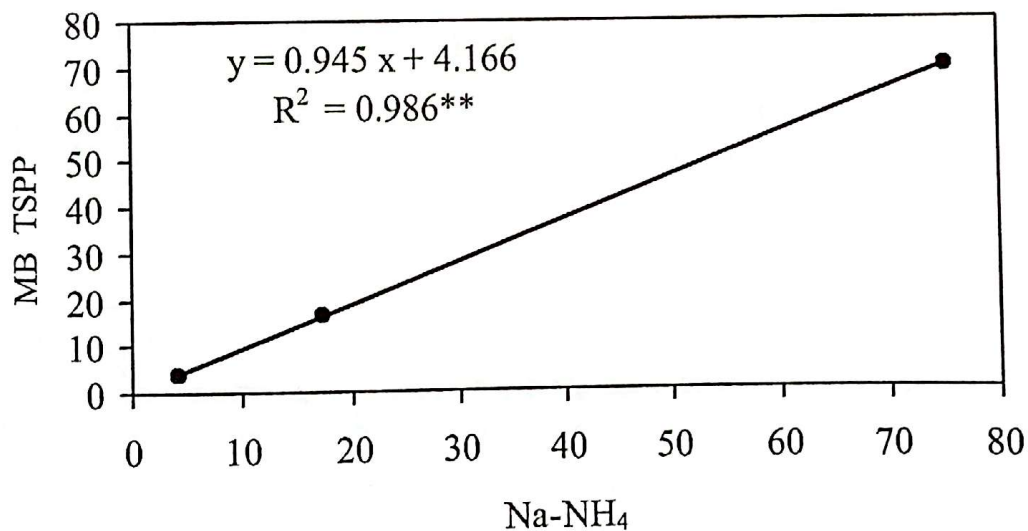


Fig. (3). Relationship between Na-NH₄ and MB-TSPP methods for determination of CEC (me100g⁻¹) in pure clay minerals.

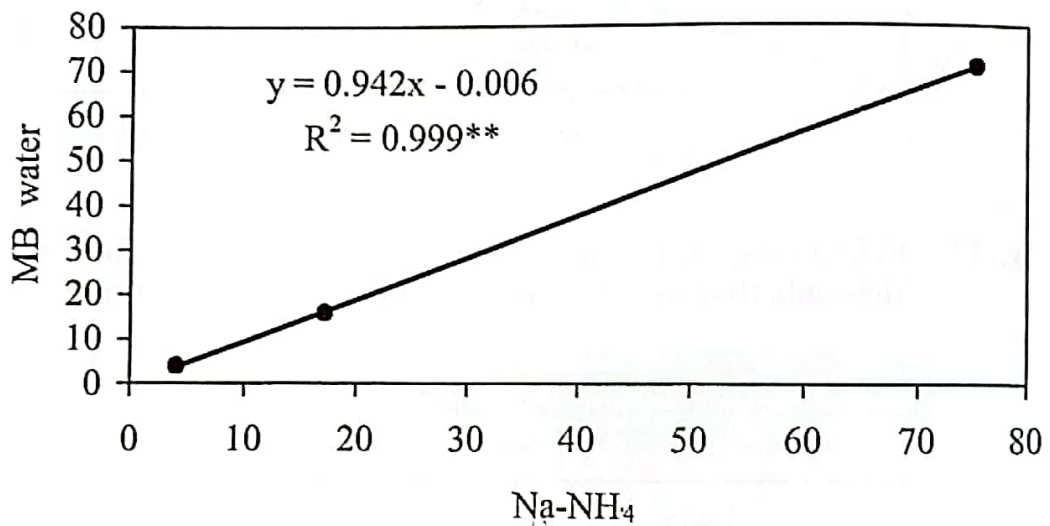


Fig. (4). Relationship between Na-NH₄ and MB-water methods for determination of CEC (me100g⁻¹) in pure clay minerals.

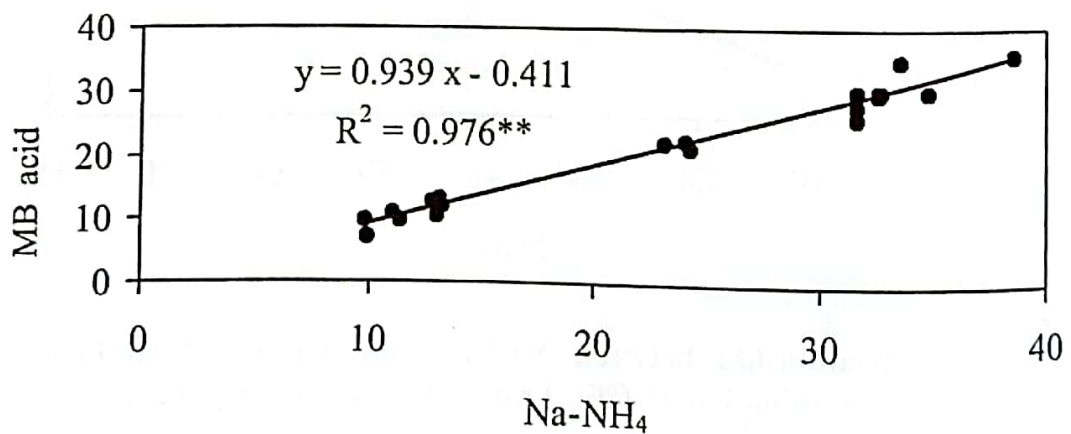


Fig. (5). Relationship between Na-NH₄ and MB-acid methods for determination of CEC (me100g⁻¹) in soil samples from Egypt.

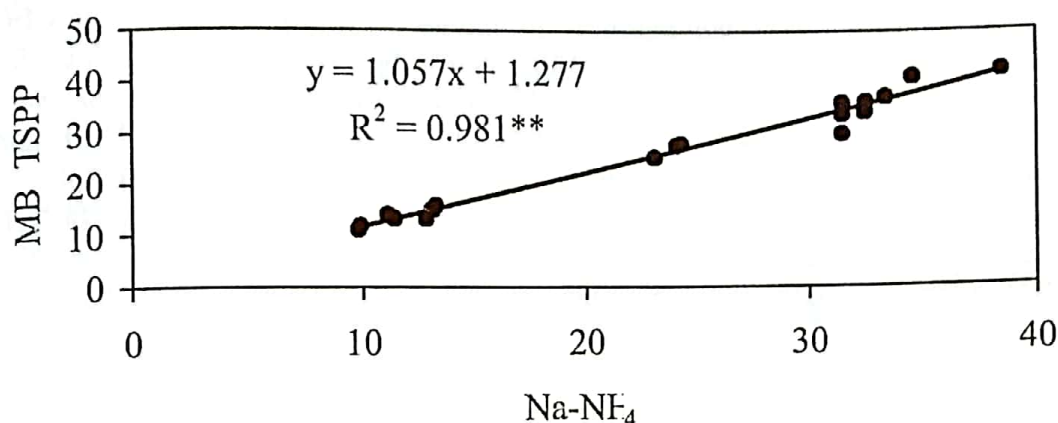


Fig. (6). Relationship between Na-NH₄ and MB-TSPP methods for determination of CEC (me100g⁻¹) in soil samples from Egypt.

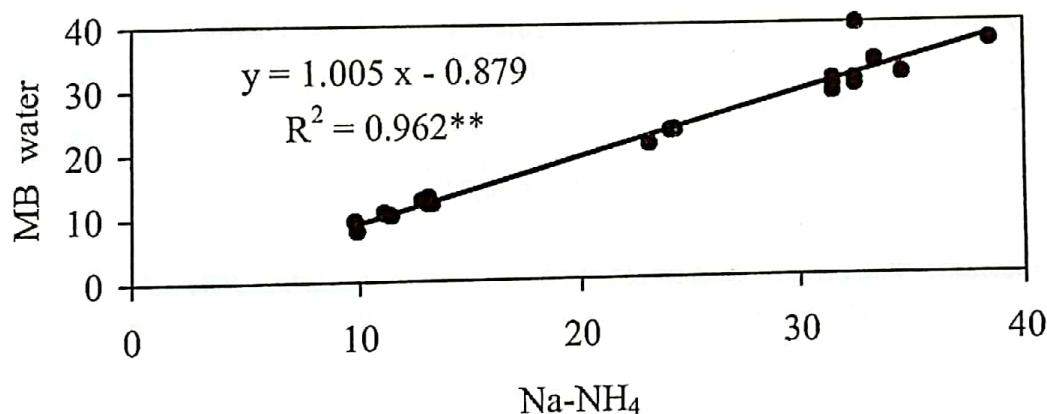


Fig. (7). Relationship between Na-NH₄ and MB-water methods for determination of CEC (me100g⁻¹) in soil samples from Egypt.

The amounts of Ca in NaOAC leachate of Burg El-Arab and Wadi El-Tumilat calcareous soils tended to be underestimated. Calcium ions dissolved from CaCO₃ inhibited completely the saturation of exchange sites with Na⁺ ions; which resulted in low CEC values. This result indicates that the reagent-grade CaCO₃ failed to absorb MB. Soon (1988) and Wang *et al.* (1996) pointed out that the CEC values of samples treated with H₂O₂ to remove organic carbon content and those of the untreated samples were not significantly different when measured by MB-acid; MB-TSPP and MB-water titration methods.

The squared regression coefficient (R^2) of MB-TSPP vs. Na-NH₄ exchange methods for soil samples is 0.98. The (R^2) of MB-water vs. Na-

NH₄ exchange methods for pure clay minerals is 1.0 (Figs 2 to 7). The MB-test is a simple, rapid, and economical method to determine CEC. The CEC values measured by MB-TSPP were closed to the calculated values for different Egyptian soils. But the values determined by MB-water were closed to the calculated values for pure clay minerals. The MB-TSPP and MB-water exchange methods may be suitable for the field testing of soils.

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موثوقية طرق تبادل أزرق الميثيلين لتقدير السعة التبادلية الكاتيونية لأراضي مصرية مختلفة ومعادن طين نقية

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* قسم كيمياء وطبيعة الأراضي - مركز بحوث الصحراء - المطرية - القاهرة - مصر .

أجريت هذه الدراسة لتقييم ومعرفة مدى دقة طريقة تبادل أزرق الميثيلين كطريقة سريعة لتقدير السعة التبادلية الكاتيونية للأراضي المصرية في مواقع مختلفة لها مدى واسع من الاختلاف في الصفات الطبيعية والكيميائية مثل قوام وبناء الأرض ومحتواها الكلي من كربونات الكالسيوم وكذلك المادة العضوية والسعة التبادلية الكاتيونية بالإضافة إلى ثلاث معادن طين نقية وهي: المونتموريللونيت المشبع بالصوديوم، الباليجورسكيت ، الكاولينيت .
جريت أربع طرق لتقدير السعة التبادلية الكاتيونية للأراضي ولمعادن الطين النقية وكانت هذه الطرق : الطريقة التقليدية (القياسية) لتبادل خلاص الصوديوم، خلاص الأمونيوم بالإضافة إلى ثلاث طرق لمعايرة تبقي ورق الترشيح وهي (MB-acid) ، (MB-TSPP) ، (MB-water) مع الأخذ في الاعتبار ظروف التركيز المناسب وفترة الاتزان وتجمع محاليل أزرق الميثيلين .
كان مربع معامل الانحدار (R^2) لطريقة MB-TSPP مقابل الطريقة القياسية لتبادل $Na-NH_4$ (٠.٩٨) وأيضاً كان (R^2) لطريقة MB-Water مقابل الطريقة القياسية لتقدير CEC وذلك لمعادن الطين النقية (١.٠) وأظهرت الدراسة أن اختبار أزرق الميثيلين بسيط وسريع واقتصادي . وكانت القيم المقدرة للسعة التبادلية الكاتيونية بطريقة MB-TSPP جيدة ومحكمة لحساب قيم السعة التبادلية الكاتيونية لمختلف الأراضي المصرية وطريقة MB-Water محكمة لحساب قيم CEC لمعادن الطين النقية وبالتالي كانت طرق MB-TSPP ، MB-Water مناسبة للتقدير الحقل للأراضي ومعادن الطين النقية ، علي التوالي .