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Research Article
Comparison of 3 Different Methods of CEC Determination in Nigerian Savannah Soils

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Abstract
Background and Objectives: Savannah soils are mainly coarse textured and thus mostly porous and dry. Cation exchange capacity varies according to differences in pH, organic matter and soil texture. Due to the possible overestimation of results with the use of NH4OAc (pH 7), there arises the need to compare and explore the results that could be obtained with the use of other methods on Nigerian savannah soils. **Materials and Methods:** Eighty samples were collected from 4 different Savannah zones, Derived Savannah, Sudan Savannah, Northern Guinea Savannah and Southern Guinea Savannah. The different method of determination used were NH4OAc (pH 7), NH4OAc (at soil pH) and BaCl. The obtained results was subjected to analysis of variance by using JMP software. Means separated by using Fishers protected least significant difference. **Results:** Results obtained using Bacl2 was generally similar to the range obtained with the use of NH4OAc (7) from soils of the savannah region. The use of NH4OAc (soil pH) gives good result on acidic soils with low content of organic matter. **Conclusion:** The amount of CEC observed with the use of different methods is influenced by the experimental conditions as well as the physicochemical properties of the soil and the exchange ability of the reagent used.

Key words: Soil analysis, Cation Exchange Capacity (CEC), ammonium acetate, barium chloride, exchangeable cations, Savannah soils, pH

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Data Availability: All relevant data are within the paper and its supporting information files.

INTRODUCTION

The quantification and characterization of the soil cation exchange capacity is of agricultural and environmental importance[1] with different methods been in use for its assessment[2-4]. With others, the exchange reaction could be carried out with use of un-buffered salts, ammonium chloride or barium chloride[5,6]. Different methods including use of buffered solutions such as; ammonium acetate, sodium acetate or barium chloride[6-8]. The major underlying principles in most of the methods involve the saturation of the soil sample with an index cation and obtained results is depends upon the nature of the cation, pH of extraction solution and initial soil pH[9]. Organic matter, iron and aluminum and clay content affects cation exchange capacity and charge density. Mostly methods for cation exchange capacity implies the cations were extractable although sometimes there could be release from non-exchangeable source as with the dissolution of Ca from carbonates[10,11]. The methods used may be grouped into determination of CEC by summation of exchangeable cations, measurement of CEC at the soil pH (effective CEC), measurement of CEC at a given buffered pH, measurement of CEC at the pH for which the charge is zero (Zero Point Charge (ZPC) or pH0)[3,5].

The determination of Cation Exchange Capacity (CEC) is an important tool in studies of erosion, retention of pollutants and waste, characterization and study of soil fertility and has wide applicability in soil mechanics[12,13]. Of all the methods developed for the determination of CEC, ammonium acetate (pH 7) the most widely used, although use of other reagents on soils have been reported such as; the use of cobalt hexamine chloride and $BaCl$[3,5,6,8]. However, there have been a lot of concerns about its use on Savannah soils due to the possible over/underestimation of results due to accumulation of calcium and magnesium salts[4,5,14]. This work was carried out to compare different methods of determination and observe which may be best suited for Savannah soils.

MATERIALS AND METHODS

Soil was sampled from 4 different Savannah regions in Nigeria, Derived Savannah, Southern Guinea Savannah, Northern Guinea Savannah and Sudan Savannah. Sampling was carried out in the year 2018. In each zone, 2 different soil orders were sampled at a depth of 0-20 cm and at 1 km interval. A total of 80 samples were collected.

Laboratory method: Basic soil parameters such as; particle size distribution was determined by using the Bouyoucos-hydrometer method following dispersion of the soil with calgon solution[15]. The soil pH was measured in both water and KCl (Park Scientific, 98%) at a soil: water and soil KCl ratio of 1:2.5 by using a glass electrode pH meter.

Ammonium acetate method: The ammonium acetate method was used as described by Ciesielski *et al.*[4] and Aprile and Lorandi[12]. In a plastic bottle, 5 g of soil was placed and 50 mL of 1N ammonium acetate added (Park Scientific, MV 74.55). The sample was shaken for 30 min, after which it was filtered and the concentrations of Mg, Ca, Na and K were read from the filtrate using AAS (Agilent technologies 200 series AA, model 240SS) and Flame photometer (Jenway, model pfp7).

Ammonium acetate method (pH): Similar to the above, but the ammonium acetate method was prepared with the pH of each of the soil sample. The sample was shaken for 30 min, after which it was filtered and the concentrations of Mg, Ca, Na and K were read from the filtrate by using AAS and Flame photometer.

Barium chloride method: About 2.5 g of sample was placed in a centrifuge and 30 mL of 1M $BaCl_2$ added. Extraction was carried out by shaking for 1 h and solid and liquid phases were separated by centrifugation at 3000 g for 10 min. The concentration of Mg, Ca, Na and K were read from the supernatant.

Data analysis: Descriptive statistics was first used to summarize the data for soil properties across the different regions and methods of determination. The analysis of variance was done by using JMP software. Means with significant differences at $p<0.05$ were separated by using Fishers protected least significant difference. Pearson's correlation was done to visualize the association between the all variables according to the different methods and across all methods.

RESULTS

Determination of physicochemical properties: The results of the physicochemical properties of the studied area are as shown in Table 1. The pH was observed to range from slightly acidic to moderately acidic. Sandy loam was observed to be the dominant soil fraction in all the soils considered with the exception of vertisols where the soils where loam in texture.

Determination of Cation Exchange Capacity (CEC): The distribution of CEC across the Savannah zone is as shown in Table 2. From the data, it was observed that the highest mean

Table 1: Physicochemical properties of soil

	pH (water)		pH (KCl)		Sand		Silt		Clay	
Location	Range	Mean	Range	Mean	Range	Mean	Range	Mean	Range	Mean
Alfisols DS	4.31-6.98	5.37	3.84-5.79	4.62	58.88-76.88	68.28	8.56-22.56	14.16	14.56-22.56	17.56
Alfisols NGS	5.60-6.61	6.16	5.48-6.20	5.83	36.88-78.88	63.68	6.56-36.56	17.16	14.56-28.56	19.16
Alfisols SGS	5.08-6.05	5.60	3.93-5.31	4.66	50.40-71.12	59.30	23.28-39.28	30.28	5.60-16.3	10.42
Alfisols SS	4.47-6.62	5.73	4.40-6.30	5.50	44.40-70.40	56.89	25.28-43.28	35.26	4.32-14.32	7.85
Inceptisols SS	4.86-6.91	5.38	3.84-4.83	4.35	52.40-74.40	64.96	13.28-21.28	16.86	12.32-28.32	18.18
Inceptisols NGS	5.27-6.48	5.67	4.70-5.45	5.09	33.12-79.12	58.79	12.56-31.28	20.25	5.60-46.32	20.96
Ultisols	4.84-5.76	5.20	4.36-5.24	4.77	70.88-84.88	81.08	0.56-16.56	4.96	12.56-16.56	13.96
Vertisols	4.72-6.83	5.64	4.01-5.19	4.76	38.88-54.88	47.48	20.56-46.56	30.16	14.56-30.56	22.36

Table 2: Distribution of CEC in Savannah soils

Method	Range	Mean	Variance	Std. Err.	CV
NH_4OAC	1.08-21.39	4.84	13.75	0.24	76.65
NH_4OAC-pH	0.95-11.65	3.74	9.28	0.20	81.51
$BaCl_2$	1.32-15.42	6.18	8.92	0.19	48.32
Region					
Derived Savannah (DS)	0.95-21.39	6.96	20.78	0.34	65.46
Northern Guinea Savannah (NGS)	1.18-13.86	4.01	6.91	0.20	65.62
Southern Guinea Savannah (SGS)	1.31-13.93	3.82	5.16	0.17	59.54
Sudan Savannah (SS)	1.32-15.21	4.89	7.55	0.20	56.22
Soil order					
Alfisols DS	0.95-21.39	5.33	25.91	0.54	95.49
Alfisols NGS	1.18-12.39	4.13	7.79	0.29	67.62
Alfisols SGS	1.32-10.15	3.18	2.88	0.18	53.27
Alfisols SS	1.75-9.65	3.57	2.71	0.17	46.17
Inceptisols SS	1.32-15.21	6.21	8.94	0.32	48.16
Inceptisols NGS	1.29-13.86	3.88	6.08	0.26	63.47
Ultisols	1.31-13.93	4.45	6.70	0.27	58.20
Vertisols	1.08-15.42	8.60	10.49	0.34	37.67

CEC: Cation Exchange capacity

Table 3: Results of CEC from different soil orders in the Savannah region as affected by the method of determination

	NH_4OAC				NH_4OAC-pH				$BaCl_2$			
Region	Range	Mean	SE	CV	Range	Mean	SE	CV	Range	Mean	SE	CV
Derived Savannah (DS)	1.08-21.39	8.15	0.72	68.48	0.95-11.63	5.42	0.54	76.48	2.04-15.42	7.33	0.42	44.79
Northern Guinea Savannah (NGS)	2.86-4.62	3.55	0.07	14.64	1.18-3.56	2.05	0.09	34.67	2.18-13.86	6.42	0.41	49.71
Southern Guinea Savannah (SGS)	2.74-4.74	3.44	0.07	15.71	1.31-9.32	2.31	0.15	51.47	2.13-13.93	5.70	0.36	49.38
Sudan Savannah (SS)	1.48-15.21	4.21	0.38	70.54	2.29-11.65	5.16	0.38	56.63	1.32-9.65	5.28	0.28	41.54
Soil Order (SO)												
Alfisols DS	2.32-21.39	7.56	1.23	88.92	0.95-1.95	1.50	0.06	20.81	2.04-12.94	6.93	0.61	48.34
Alfisols NGS	2.86-4.41	3.65	0.09	13.92	1.18-3.08	1.98	0.12	32.71	2.18-12.39	6.75	0.61	49.47
Alfisols SGS	2.74-3.59	3.17	0.04	7.75	1.32-2.74	1.85	0.08	23.01	2.13-10.15	4.53	0.40	48.66
Alfisols SS	1.75-3.62	2.88	0.11	20.64	2.29-3.79	2.96	0.10	18.38	2.42-9.65	4.85	0.41	46.58
Inceptisols SS	1.48-15.21	5.55	0.68	67.38	2.88-11.65	7.37	0.49	36.14	1.32-8.53	5.71	0.38	36.30
Inceptisols NGS	2.90-4.62	3.45	0.09	15.06	1.29-3.56	2.12	0.14	36.54	3.08-13.86	6.09	3.06	50.20
Ultisols	2.97-4.74	3.71	0.11	16.80	1.31-9.32	2.77	0.27	54.11	2.60-13.93	6.86	2.90	42.33
Vertisols	1.08-14.85	8.73	0.76	47.75	5.39-11.63	9.34	0.32	18.79	2.40-15.42	7.72	3.22	41.68

CEC: Cation Exchange capacity

of 6.18 cmol kg^{-1} was observed with the use of BaCl with a CV of 48.32, which was followed by NH_4OAc with a mean of 4.84 cmol kg^{-1} and CV of 76.65 and lastly by NH_4OAc-pH which have a mean of 3.74 cmol kg^{-1} and CV of 81.51.

In all the regions under study, the Derived Savannah (DS) showed the highest result with a mean of 6.96 cmol kg^{-1} and CV 65.46 of followed by the Sudan Savannah (SS) which showed a mean of 4.89 cmol kg^{-1} and CV of 56.22. The lowest result of CEC (3.82 cmol kg^{-1}) was observed in the southern Guinea Savannah (GS).

It was also observed from the data that the highest CEC (8.60 cmol kg^{-1}) was obtained from Vertisols in the DS followed closely by the Inceptisols from the Sudan Savannah zone with a mean of 6.21 cmol kg^{-1}. The lowest CEC content

Table 4: Mean comparison of CEC by using different methods and in different soil orders and region

Method (M)	CEC (cmol kg^{-1})
NH_4OAc	4.84[b]
NH_4OAC-pH	3.74[c]
$BaCl_2$	6.18[a]
SE±	
p-value	<.0001
Soil Order (SO)	
Alfisols DS	5.33[c]
Alfisols NGS	4.13d[e]
Alfisols SGS	3.18[f]
Alfisols SS	3.57[ef]
Inceptisols SS	6.21[b]
Inceptisols NGS	3.88d[ef]
Ultisols	4.45[d]
Vertisols	8.60[a]
SE±	0.269
p-value	<.0001
Region	
Derived Savannah (DS)	6.96[a]
Northern Guinea Savannah (NGS)	4.01[c]
Southern Guinea Savannah (SGS)	3.82[c]
Sudan Savannah (SS)	4.89[b]
SE±	0.218
p-value	<.0001
Interaction	
R*M	**
SO*M	**

Means followed by different letters are significantly different at $p<0.05$, **Significant interactions ($p<0.001$), CEC: Cation Exchange capacity

Table 5: Correlation of CEC with soil physical properties for each method of detection

Parameters	NH_4OAc	NH_4OAc-pH	$BaCl_2$
pH (KCl)	0.04	0.28***	0.03
pH (H_2O)	0.02	0.33***	-0.10
O.C	0.08	0.31***	-0.02
Sand (%)	-0.11	-0.47***	-0.15*
Clay (%)	0.20**	0.45***	0.07
Silt (%)	-0.01	0.23***	0.12

*,**,***p-values <0.05, 0.01 and 0.001, respectively, CEC: Cation Exchange capacity

of 3.18 cmol kg^{-1} was observed from the Alfisols of the southern Guinea Savannah. The CV observed was high (>35).

The results of CEC from different soil orders and Savannah region as affected by the method of determination is as presented on Table 3. It was observed in the derived Savannah that the highest CEC content of 8.15 cmol kg^{-1} was obtained by using NH_4OAc followed by use of Barium Chloride 7.33 cmol $kg^{-1}$and lastly by NH_4OAC-pH with a mean of 5.42 cmol kg^{-1}. In the northern Guinea Savannah, the highest result of 6.42 cmol kg^{-1} was observed with the use of BaCl followed by NH_4OAC with a mean of 3.55 cmol kg^{-1} and lastly NH_4OAc-pH having a mean of 2.05 cmol kg^{-1}. The observed CV was moderate in both NH_4OAc and NH_4OAc-pH (CV<35) and high with the use of BaCl CV>35. Similar trends were observed in the southern Guinea Savannah except that the observed CV was moderate only in NH_4OAc. The Sudan Savannah showed a very high results with the use of BaCl for CEC measurements with mean of 5.28 cmol kg^{-1} followed by NH_4OAc-pH with a 5.16 cmol kg^{-1} mean of and lastly by NH_4OAc with a mean of 4.21 cmol kg^{-1}. Observed CV was high regardless of the method used (CV>35).

The highest value of 7.56 cmol kg^{-1} was observed in the Alfisols (DS) with the use of NH_4OAc followed by BaCl with a mean of 6.93 cmol kg^{-1} and the lowest results of 1.50 cmol kg^{-1}observed with NH_4OAc-pH. In the Alfisols (NGS), use of BaCl gave the highest result of 6.75 cmol kg^{-1} which was followed by NH_4OAc with a mean value of 3.65 cmol kg^{-1} and then by NH_4OAc-pH with a mean of 1.98 cmol kg^{-1}. Similar results were observed in the Alfisols (SGS), Inceptisols (NGS) and Ultisols. The results observed in the Alfisols (SS) was 4.85, 2.96 and 2.88 cmol kg^{-1}, the use of BaCl, NH_4OAc-pH and NH_4OAc, respectively. In the Inceptisols of the Sudan Savannah, the highest result of 7.37 cmol kg^{-1} was observed by NH_4OAc-pH followed by BaCl with a mean of 5.71 cmol kg^{-1} and then by NH_4OAc with a mean of 5.55 cmol kg^{-1}. The trends was similar to what was observed in the Vertisols, although the results obtained by NH_4OAc were higher than BaCl. Generally, the observed CV with NH_4OAc across all the soil orders ranged from low to high (7.5<CV>88.92), while, BaCl was high, CV>35 and NH_4OAc-pH was moderate to high.

The results of the mean comparison of CEC by using different methods and in different soil orders and region are as presented on Table 4. The results showed statistical difference ($p<0.05$) among the different methods. $BaCl_2$ had the highest result of 6.18 cmol kg^{-1} followed by 4.84 cmol kg^{-1} of NH_4OAc and then NH_4OAc-pH with a value of 3.74 cmol kg^{-1}. Among the different soil orders, a significant difference ($p<0.05$) was observed. The highest result of 8.60 cmol kg^{-1} was observed in the Vertisols followed by 6.21 cmol kg^{-1} in the Inceptisols (SS) and 5.33 cmol kg^{-1} in the Alfisols (DS). Alfisols (SGS) was observed to have the lowest value of 3.18 cmol kg^{-1} although the observed results were statistically similar to what was obtained in the Alfisols (SS) and the Inceptisols (NGS). A degree of difference was also observed across the different regions ($p<0.05$). The highest result was obtained in the Derived Savannah with the lowest result observed in the southern Guinea Savannah.

Highly significant interactions ($p<0.001$) was observed between the different methods and soil orders as well as between the different methods and Savannah regions.

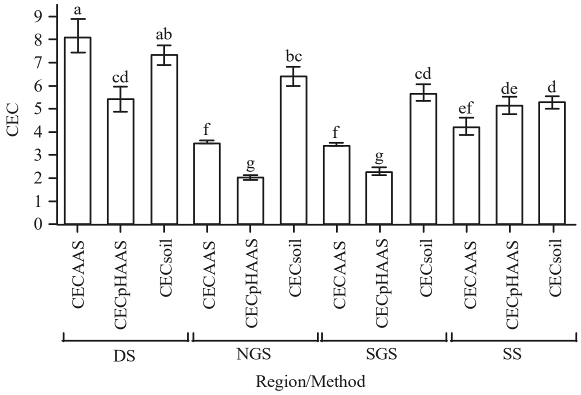

Fig. 1: Interaction between agro-ecological region and method of detection of CEC

Error bars are standard error of mean, different alphabet for each bar represents significant mean difference at $p<0.05$, CEC: Cation exchange capacity

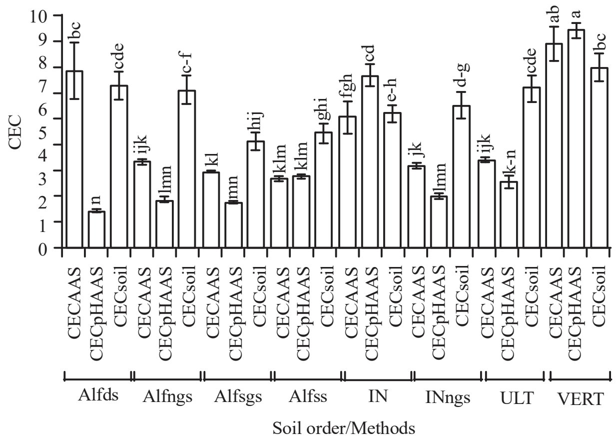

Fig. 2: Interaction between soil order and method of detection of CEC

Error bars are standard error of mean, different alphabet for each bar represents significant mean difference at $p<0.05$, CEC: Cation exchange capacity

Table 6: Correlation of CEC for each method of detection in different regions

Parameters	NH_4OAc	NH_4OAc-pH	$BaCl_2$
NH_4OAc	1		
NH_4OAc-pH	0.33***	1	
$BaCl_2$	0.20**	0.11	1

,*p-values <0.01 and 0.001, respectively, CEC: Cation Exchange capacity

Figure 1 shows the interaction between agro-ecological region and method of CEC determination. The data sowed that the best results was obtained in the Derived Savanah with the use of NH_4OAc which was statistically similar with the use of BaCl. The least results was obtained with the use of NH_4OAc-pH in both the northern Guinea Savannah and the southern Guinea Savannah.

The results of the interaction between soil order and different methods of CEC determination is as shown in Fig. 2. From the data, it was observed that the best results was obtained with the use of NH_4OAc-pH in Vertisols and the results were statistically similar to use of NH_4OAc in same soil. The use of BaCl for determination also gave a statistically similar results in the same Vertisols as with use of NH_4OAc in the Alfisols (DS). The lowest result was obtained with the use of NH_4OAC-pH in the Alfisols of the Derived Savannah.

Table 5 shows the pairwise correlation of soil physical properties with CEC determination by different methods. A highly significant ($p<0.001$) and positive correlation were observed between use of NH_4OAc and clay content ($r = 0.20^{**}$). Use of NH_4OAc-pH showed a positive and highly significant correlation with all properties except sand where a negative correlation was observed ($r = -0.47^{***}$). The association between use of BaCl and physical properties only showed a negative and significant relationship with sand ($r = -0.15^{*}$).

Table 6 shows the pairwise correlation between the different methods of CEC determination. From the data, it was observed that NH_4OAc had a highly significant ($p<0.01$ and $p<0.001$) and positive correlation with NH_4OAc-pH and $BaCl_2$ ($r = 0.33^{***}$ and $r = 0.20^{**}$), respectively

DISCUSSION

The majority of the study sites were dominantly sandy loam in texture with higher fraction of sand thus, Akpa *et al.*[16] and Akinbola *et al.*[17] had suggested that the high sand fraction in most Savannah soils could be related to the granitic origin of the parent materials and alleviation identified as the chief factor responsible for its removal. The high sand fraction could also be attributed to the geological processes involving sorting of soil materials by biological activities as well as the interplay of soil forming factors as reported by Malgwi *et al.*[18] and Tanko[19]. The acidic nature of the soils could be related to the nature of fertilizers that have been applied over time as application of nitrogenous fertilizer in the form of inorganic fertilizer have been reported to lower will lower pH and accelerate acidity in soils which is in agreement with the work of Jimoh *et al.*[20].

The CEC of Savanna soil have been rated low to medium due to the prevalence of low activity clays and low organic matter content[21,22]. The results obtained where within the expected limits regardless of the method, region or soil order. Use of different methods for same soil has been known to produce different results[23] and these differences may be as a

2result of the different reactions induced by extract ants which may vary according to soil properties; pH, organic matter, texture and clay[8,14]. The results obtained with the use of $BaCl_2$ could be related to its capacity for salt dissolution during extraction[2,3]. The rate of extraction of $BaCl_2$ could also be related to the large volume of the reagent used indicating the results obtained might be influenced by the quantity of the reagent used rather than the reagent itself[2,3,6]. However, Bouyoucos *et al*.[14] has explained that high results by $BaCL_2$ could also be due to its low ionic strength and closeness to soil natural pH.

Use of NH_4OAc also gave good results regardless of the order or region which could be due to its acidification of the soil resulting in the solubility of more cations[8]. However, the quantity of the solubilized cations is usually dependent on the concentration of cations present in the soil samples[6]. The use of NH_4OAc-pH at measured soil pH yielded relatively good results and the research explained that the buffered pH may preserve the specificity of the exchange reaction by non-modification the soil and the non-pH variations in soil resulting in the elimination of negative charges on clay minerals and organic matter by dissociation of weak acid groups[2,3].

The general results was observed in the order $BaCl>NH_4OAc>NH_4OAc$-pH which was similar to the trends observed by Jaremko and Kalembasa[7] and Bouyoucos *et al*.[14] although sodium acetate (pH 8.2) was used. However, the trend observed in some results was in the order NH_4OAc-pH>NH_4OAc>BaCl as in the case of Vertisols which showed the highest CEC amongst all the soil which may be related to the soil's composition, acidic, moderate levels of organic carbon and clay content[7] as well as the nature and quantity of the index cation used (NH_4OAc-pH) which could have had an influence on the reaction yield[4,13]. It may also have been due to a balanced extraction of the exchangeable cations at the soil's native pH[24].

The positive and high correlation observed between CEC and organic carbon have been related to the dissociation of organic acids resulting in the formation of negative charges, thereby increasing affinity for cations[23]. The positive association with clay and pH has also been related to the fraction's ability adsorb and hold onto cations while pH increase the negatively charged sites and consequently the CEC[25].

Correlation between methods was low with significant differences observed between the methods. Difference in the methodology between the different methods could be connected with the pH of extract solution and soil[13]. The CEC, NH_4OAc-pH and $BaCl_2$ is a measure of soil CEC at "field" pH (effective CEC), while NH_4OAc is a measure of soil CEC buffered at pH = 7, which may result in highly inflated values for CEC[3,13]. Apart from displacement of cations with the use of NH_4OAc, there could also be a displacement of exchangeable acidity equivalent to the amount of alkali required to bring the soil from the field pH to pH 7[13].

Generally, using of summation method as done in this current study has been found to give a very good estimation of CEC as it gives a value close what is obtained on the field and especially very reliable on soils with pH<7 [1,3,13].

CONCLUSION

The concentration of cations obtained with each method is observed to be influenced by physicochemical conditions properties of the soils, exchange ability and quantity of index cation used. Among all the methods used, $BaCl_2$ was observed to give better and almost similar results with NH_4OAc (pH 7). Use of NH_4OAc (at soil pH) was less comparable to NH_4OAc (pH 7) and BaCl although its use showed very good results on Vertisols. NH_4OAc (at soil pH) had positive relationship with organic carbon, pH and clay. Hence, it can be concluded from this work that all the three methods used are not comparable and further need to explore use of other methods.

SIGNIFICANCE STATEMENTS

This study observed that the use of different methods on same type of soil produce significantly different results which has been attributed to the differences in the chemical composition of the reagents. Use of NH_4OAc (at soil pH) could give very good results in acidic soils with low moderate levels of organic carbon and clay. This study showed that use of other methods yield good results for CEC and hence, the need for exploration of methods that can determine the CEC close to native soil pH and give comparatively good results.

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