

Development of low cost soil testing methods for sulphur and calcium

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ABSTRACT

To develop methods for testing soil sulphur (S) and calcium (Ca) as a low cost technology for fertilizer recommendation, a study was undertaken in the Department of Soil Science at Bangladesh Agricultural University (BAU), Mymensingh. Three extracting reagents viz. Morgan solution (NaOAc + HOAc, pH 4.8), 0.15% CaCl₂ and water, each followed by two colour developing reagents such as BaCl₂ plus FeCl₃ and BaCrO₄ were tested for determination of soil S. For the determination of soil Ca, Morgan solution and 1 M NH₄OAc (pH 7.0) accompanied with a common turbid forming reagent Na₂C₂O₄ were used. Among the extractants tested, Morgan solution was found more effective for extracting both soil S and Ca than the others. Between two colour producing reagents, BaCrO₄ produced more stable colour in acidic condition compared with BaCl₂ plus FeCl₃. In case of Ca, the performance of Na₂C₂O₄ was found quite satisfactory. In comparison to conventional laboratory methods these proposed methods for S and Ca are more economic.

INTRODUCTION

Nutrient deficiency in soil is a major factor for declining crop production in Bangladesh. Nutritional complexity in Bangladesh soils has been increasing steadily from mid 60's and deficiency of more nutrients is occurring over time. Before 1980, deficiency of N, P, and K was a major problem in Bangladesh soils and thereafter deficiency of S, Zn, B and Ca were also found in some soils (Hoque and Jahiruddin, 1994). Recently, the deficiency of those nutrients is spreading to more soils of the country mainly due to intensive cropping with modern varieties. Sadat (2000) reported that the status of S was very low to low and calcium was low to medium in six different AFZs of northern areas of the country. Ahmed (2002) also found severe deficiency of S in some soils of Bangladesh.

Fertilizers are now-a-days, indispensable for crop production in modern agriculture. Among the factors that influence crop production, only fertilizer can contribute 50% of the total increase.

The fertilizers should be used in a balanced way else the continuous unbalanced use of fertilizers may deteriorate the yield as well as soil fertility. As soil is a heterogeneous body and its physical, chemical and biological processes are dynamic, one general fertilizer recommendation could not be synchronous with the requirement of crops for all nutrients as well as for all soils. Because of unavailability of soil testing facilities, most of the farmers are using fertilizers blindly and indiscriminately. This implies the necessity of soil fertility evaluation and balanced fertilization for sustaining soil health and crop yield. The Government of Bangladesh has given a great emphasis on agricultural development of the country in order to feed its ever-increasing population. An attention has been paid to soil test based fertilizer used for sustaining the soil resources of the country and for improving the crop yield and quality.

Standard laboratory based soil testing methods can give accurate results, however the cost of the test is comparatively high and further the method is

time consuming. Most of the farmers in this country cannot afford the cost of soil test. Considering this point in view, the Department of Soil Science, Bangladesh Agricultural University, Mymensingh has developed a Soil Testing Kit as a means of low cost soil testing facility. It is now being used at farmer's level. Methods for testing NPK and soil pH have been included in the BAU, Soil Testing Kit. Now there is a necessity of including methods for testing soil S and calcium. The advantage of the use of Soil Testing Kit is that, the method is simple, rapid and cheap with a reasonable result. With this end in view, the present work was undertaken to develop low cost soil testing method for S and Ca for fertilizer recommendation at farmer's level.

MATERIALS AND METHODS

The work was conducted at the Soil Testing Laboratory in the Department of Soil Science, BAU, Mymensingh to develop methods for rapid determination of soil S and calcium for inclusion in the BAU Soil Testing Kit.

Soil sample collection and laboratory analysis

The soil samples were collected from Patuakhali, Bogra, Gazipur, Mymensingh, Khulna, Comilla and Rajshahi districts. Soil samples were collected from 10 sampling sites of each district. These soils were then air dried at room temperature in the laboratory, ground and pass through 10 μm pore size mesh sieve and stored. Physical and chemical properties of the soils were analyzed in the Department of Soil Science, Bangladesh Agricultural University, Mymensingh. Particle size analysis of soil was done by Hydrometer method (Bouyoucos, 1962). The textural class was determined by plotting the percent values of sand,

silt and clay content on to the Marshall's Triangular Co-ordinate following USDA system. Soil pH was measured with the help of a glass electrode pH meter, using soil: water suspension of 1:2.5 as described by Jackson (1958). EC of equilibrium extractant solutions was measured by a conductivity meter (Page, et. al., 1982). The ionic strengths of equilibrium extractant solutions were calculated following the formula 0.013EC in m mols cm^{-2} (Lindsay, 1979). Sulphur content in the extract determined by developing turbidity of BaSO_4 with BaCl_2 solution. The intensity of turbidity was measured by a spectrophotometer at 420 nm wavelength. Exchangeable calcium of soils was determined from 1N NH_4OAc (pH 7.0) extract by Flamephotometer (Page et. al., 1982).

Determination of Soil S

Selection of extractant for soil sulphur

Available sulphur in soil was extracted with three extractants i.e. Morgan solution ($\text{NaOAc} + \text{HOAc}$, pH 4.8), CaCl_2 (0.15%) and water.

Method-1

Formation and enhancement of turbidity

Ten ml of SO_4^{2-} solution of different concentrations were taken in test vial in triplicate. One ml of BaCl_2 as turbid reagent (solution: reagent ratio=10:1) was added to the test vial. After five minutes, the turbidity developed for different concentrations was recorded with photograph. The difference between concentrations or different status such as low to very high was detected by observing the visibility of a wide dark black line through the turbid solution.

Table 1
Outline of the methods for S extraction.

Extractants	Soil: Extractant ratio		Shaking time (min)		Reference
	Standard method	Kit method	Standard method	Kit method	
0.15% CaCl_2	1:5	1:2	30	1	Page et al. (1982)
Morgan solution	1:5	1:2	30	1	Lunt et al. (1950)
Water	1:5	1:2	30	1	Dewis and Freitas (1984)

A standard solution equal to the concentration of soil solution contains a very low amount of S for developing sufficient turbidity and for differentiation between concentrations. This problem was overcome by adding FeCl_3 solution which serves as a colour producing reagent. For this purpose sulphate (SO_4^{2-}) solutions (10ml) of different concentrations were taken in a series of test vial. Ten drops of FeCl_3 solution was added, followed by one ml of BaCl_2 . Turbidity of BaCl_2 with additional colour was developed which was recorded with photograph for comparison.

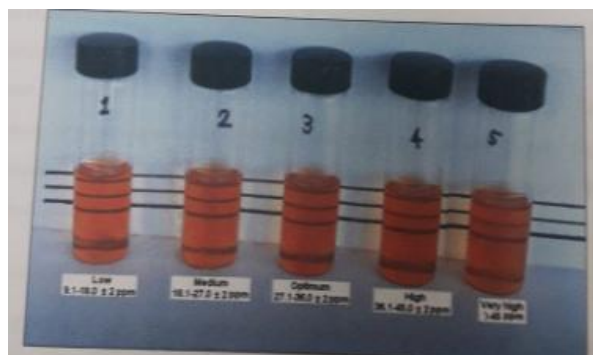


Figure 1
Colour chart for S in water for Method-1.

Preparation of colour chart and testing

Standard solutions of SO_4^{2-} viz. were prepared by dissolving K_2SO_4 in Morgan solution. The 5, 10, 15, 20 and 25 ppm solution showed distinct variation in density of turbidity from immediate lower/higher concentration. Hence, these concentrations were considered for preparation of colour chart (Figure 1). S from soil sample was extracted with different extracting solutions (0.15% CaCl_2 , Morgan solution and water maintaining soil: extractant ratio of 1:2) in order to confirm whether the proposed method can be applied to determine soil S. All the results were compared with colour chart and were found suitable.

Method-2

After precipitation of barium sulphate, the excess barium reagent is rendered insoluble by making the solution alkaline, when the dichromate ion is converted back to chromate ion. The combined precipitation of barium sulphate and barium chromate gives a yellow solution whose depth of

colour is related directly to the original sulphate concentration (Dewis and Freitas 1984).

A series of SO_4^{2-} solution of different concentrations i.e. 0, 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 12, 13, 14, 15, 16, 17, 18, 19, 20, 21, 22, 23, 24, 25, 26, 27, 28, 29 and 30 ppm were prepared by diluting the stock solution. Two ml of BaCrO_4 solution and 10 drops of NaOH were taken in a set of 30 test vials (selected for 30 concentrations). An amount of 10 ml of SO_4^{2-} solution of each concentration was added to the test vial followed by 8 drops of NaOH and the turbidity was observed. In this test, the precipitation settled quickly at the bottom of the vial leaving clear supernatant solution above. The colour variation of the supernatant solution for different concentrations was not distinct and it was difficult to differentiate the variations for different concentrations. Hence, a slight modification was made in method-II as described below.

Method-3 (Modification of Method-2)

Formation of turbidity

Sulphate solutions of different concentrations were prepared and 10 ml of these solutions was taken in a series of test tubes as used in method 2. Four drops of conc. The HCl (6 N) was added in each solution followed by 2 ml of BaCrO_4 in each test tube. Ten drops of NaOH (5 N) was added and yellowish-white turbidity was found. The colour intensity of the turbidity was proportional to the SO_4^{2-} ion concentration in the solution. A coloured photograph was made for future use.



Figure 2
Colour chart for S in water for Method-3.

Finally SO_4^{2-} solution of 5, 10, 15, 20 and 25 ppm were again prepared and reagents were added following the procedure stated above for preparing a color chart (Figure 2). S of soil samples was extracted with different extracting solution (0.15% CaCl_2 . Morgan solution and water with soil: extractant ratio of 1:2) were tested to confirm whether the method can be used to determine soil S. The results were compared with colour chart and found suitable. The depth of turbidity was detected by observing the visibility of a wide dark black line through the turbidity.

Method of determination of soil calcium

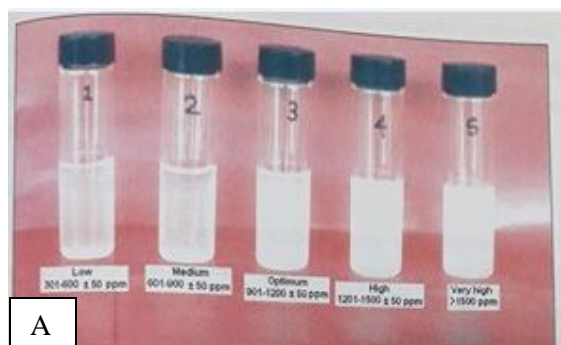
Table 2

Outline of the methods used for calcium extraction.

Extractants	Soil: Extractant ratio		Shaking time (min)		Reference
	Standard method	Kit method	Standard method	Kit method	
NH_4OAc	1:10	1:2	30	1	Page <i>et al.</i> (1982)
Morgan solution	1:10	1:2	30	1	Lunt <i>et al.</i> (1950)

Formation of turbidity and preparation of colour chart

A series of calcium solutions of different concentrations, viz. 800, 1200, 1600 and 2000 ppm were prepared by diluting 2000 ppm stock solution. An amount of 10 ml of each solution was taken in test vials followed by 1 ml of the reagent (Na-oxalate). It was shaken for mixing and after 5 minutes, the white turbidity was found in vials whose density corresponded to the concentration of calcium in solution.



Several methods have been suggested by different scientists to determine calcium in soil, plant and water samples (Lunt et al., 1950; Page et al., 1982; Jackson 1962). An attempt has been taken here to develop method for rapid determination of calcium in soils.

In general NH_4OAc (pH 7.0) is used to extract available form of most of the cations in soil. An attempt was taken to test soil with both the extractants to see whether Morgan solution can be used as a substitute of NH_4OAc for determining exchangeable calcium.



Figure 3

A. Colour chart for Ca in Morgan solution. B. Colour chart for Ca in NH_4OAc solution.

Finally an amount of 1 ml of sodium oxalate solution was added in each vial and white turbidity of calcium oxalate for 150, 300, 450, 600, 750 and 900 ppm Ca showed distinct variation in colour with each other. Hence, this concentration was selected for preparing colour chart (Figure 3).

Soil samples with varying soil pH and texture were collected from different places for confirmation test. A number of 15 test vials were

earmarked for each soil sample. These vials were divided into 5 groups for 5 different shaking times with 3 replications. As such there were 150 (15×10) test vials for 10 soil samples. An amount of 5 g of soil was taken in each of 15 test vials; 10 ml of Morgan solution was added in each vial and was shaken for different times as stated above. After shaking, they were filtered and 5 ml extract was collected in another test vial with funnel previously marked to 5 ml. an amount of 0.5 ml (10 drops) of turbidity reagent was added to each vial containing 5 ml extract and the turbidity thus developed after 5 minutes was compared with photograph taken previously.

RESULTS AND DISCUSSION

Extraction of available S from soil

Figure 4-7 represents the results of available S concentration of 10 selected samples of varying properties extracted by 3 different extractants. It appears that the amount of extracted S varied considerably between soils and extractants (Alam et al., 2005). The extracting agent 0.15% CaCl_2 was taken as standard for comparing the effectiveness of Morgan solution and water as extracting solution. In general, Morgan solution extracted the highest amount of S followed by 0.15% CaCl_2 and water (Fig. 14-17). Thus, Morgan solution can be regarded as the best extractant for soil S. Sodium is a dispersing agent and it thus, dispersed the particles when added to a soil creating more facilities for higher soil solution interaction. When the Morgan solution was added to soil the Na^+ (ion) probably dispersed the soils as a consequence of which acetate ion replaced more SO_4^{2-} from surface of soil solids. This might be one of the causes of obtaining higher results with Morgan solution.

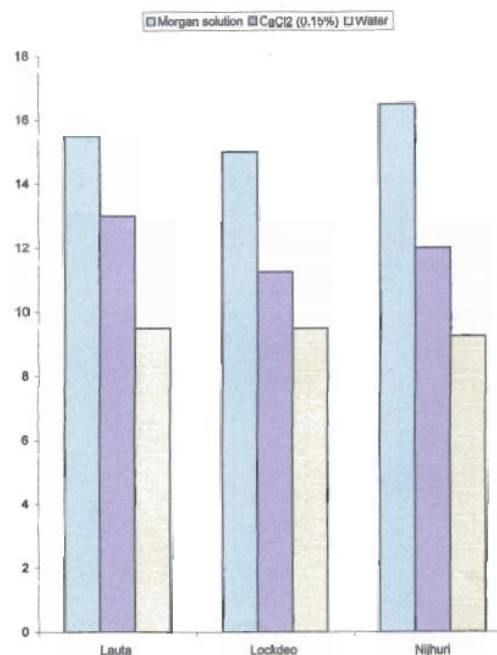


Figure 4
Sulphur status in soils of Lautla, Lockdeo and Nijhuri series extracted by three extractants.

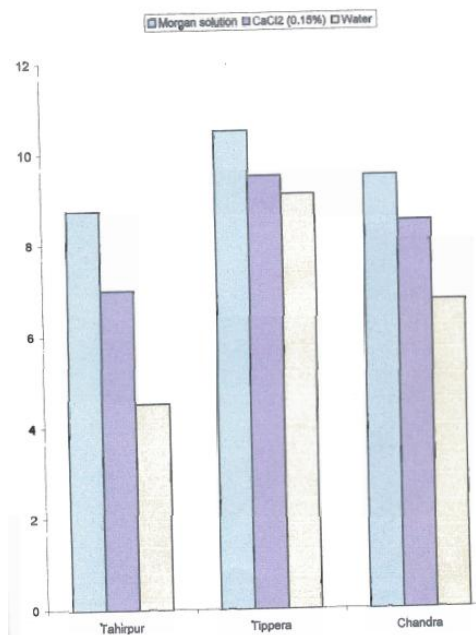


Figure 5
Sulphur status in soils of Tahirpur, Tippera and Chandra series extracted by three extractants.

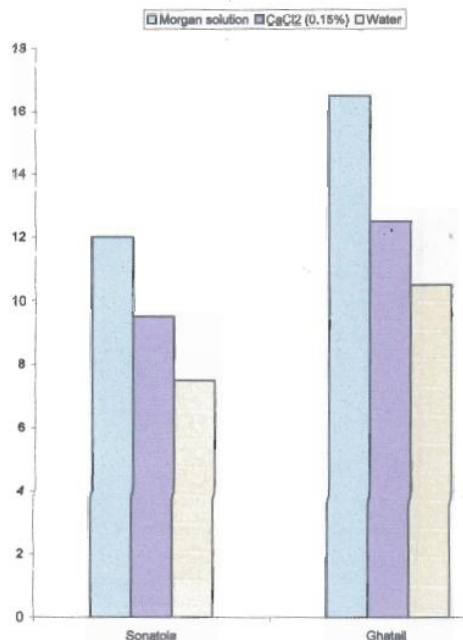


Figure 6
Sulphur status in soils of Sonatola and Ghatail series extracted by three extractants.

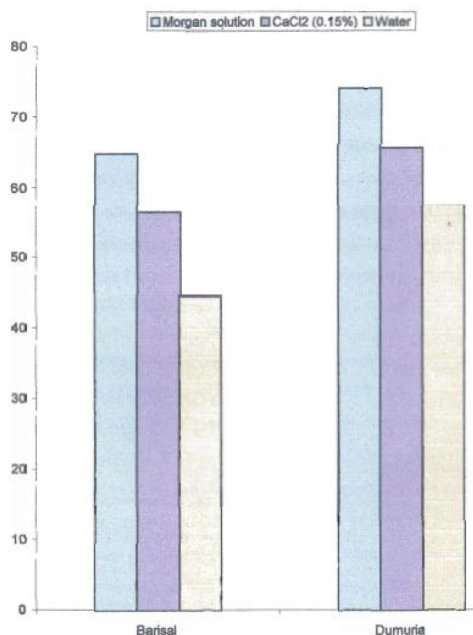


Figure 7
Sulphur status in soils of Barisal and Dumuria series extracted by three extractants

On the other hand, calcium is generally considered as the cementing agent and hence, reduces soil dispersion. It seems that when CaCl₂ (0.15%) solution was added to the soil, the soil particles

were not sufficiently dispersed and the Cl⁻ (ion) got less opportunity to replace SO₄²⁻ form exchange site. The adsorption/exchange capacity of a solution increases with increasing ionic strength. The ionic strength of Morgan solution (6.409) was almost more than 200 times higher than the CaCl₂ solution (0.02821). This higher ionic strength of Morgan solution also helped extracting more SO₄²⁻ from soil. The pH value of Morgan solution was 4.8. But the pH value of 0.15% CaCl₂ was 6.8 i.e. almost neutral. This acidity of Morgan solution possibly helped dissolving more CaSO₄H₂O in calcareous soil increasing the concentration of SO₄²⁻ in the extract compared to CaCl₂ (0.15%).

Considering the extracted SO₄²⁻ status of different soils, a wide variation from 8.75 to 74.0 ppm, 7.0 to 65.5 ppm and 4.5 to 57.5 ppm was found for Morgan solution, CaCl₂ (0.15%) and water, respectively. Soils of Ganges Tidal Floodplain i.e. Barisal and Dumuria contained very high amount of SO₄²⁻ ranging from 44.5 to 64.75 and 57.5 to 74 ppm, respectively, irrespective of extraction methods. The status of other soils varied from 4.5 to 16.5 ppm irrespective of extracting methods. This variation in sulphur status of soils was probably associated with original deposition of soil material and the management practices carried out thereafter. However, with minor exceptions the trend of soil sulphur interactions was similar for all extractants, although the magnitude varied between extractants. Hence, there was a strong relationship between the extractants (*r* values all above 0.993). It also indicated that the Morgan extracting solution was more effective than the other extractant for all types of soils.

Methods for testing sulphur in the soil extract

Method-1

The turbidity developed in this method for different concentrations was compared with the standard colour chart prepared according to the grading of BARC (1997). It appears that the grading of sulphur status i.e. very low to very high coincides very nicely with the obtained results for different extractants (Table 3). This method-1 could be suggested for rapid analysis of soil sulphur for fertilizer recommendation at farmer's

level. But it needs skillness to detect correct status of sulphur by carefully observing the visibility of dark-line through the turbid solution. It is difficult to differentiate the difference in visibility of dark-line for immediate higher and lower concentrations. The addition of FeCl_3 makes a clear difference between two concentrations in a narrow range by increasing the distinctness of dark-line through turbid.

Table 3
Status of sulphur determined by Method-1.

Soil Series	Morgan solution (NaOAc-HOAc)	CaCl_2 (0.15%)	Water
Sonatola	Low	Low	Very low
Barisal Chandra	Very high Low	Very high Very low	High Very low
Dumuria	Very high	Very high	Very high
Ghatail	Low	Low	Low
Lauta	Low	Low	Low
Lockdeo	Low	Low	Low
Nijhuri	Low	Low	Low
Taherpur	Very low	Very low	Very low
Tippera	Low	Low	Low

Table 4
Status of sulphur determined by Method-2.

Soil Series	Morgan solution (NaOAc-HOAc)	CaCl_2 (0.15%)	Water
Sonatola	Low	Low	Very low
Barisal Chandra	Very high Low	Very high Very low	High Very low
Dumuria	Very high	Very high	Very high
Ghatail	Low	Low	Low
Lauta	Low	Low	Low
Lockdeo	Low	Low	Low
Nijhuri	Low	Low	Low
Taherpur	Very low	Very low	Very low
Tippera	Low	Low	Low

Table 5
Status of sulphur determined by Method-3.

Soil Series	Morgan solution (NaOAc-HOAc)	CaCl_2 (0.15%)	Water
Sonatola	Low	Low	Very low
Barisal Chandra	Very high Low	Very high Very low	High Very low
Dumuria	Very high	Very high	Very high
Ghatail	Low	Low	Low
Lauta	Low	Low	Low
Lockdeo	Low	Low	Low
Nijhuri	Low	Low	Low
Taherpur	Very low	Very low	Very low
Tippera	Low	Low	Low

Method-2

The results of qualitative analysis of available S extracted by different extractants and analysed by selected Method-2 have been presented in Table-6. As stated under section 3.2.3., the turbidity was developed by barium chromate and sodium hydroxide under alkaline condition. This Method-2 also gave a good result for the extracted sulphur according to the BARC (1997) grading as stated above. However, the problem faced in analyzing the extracted sulphur following the Method-2 is that in alkaline condition the turbidity developed settled very quickly and it became difficult to differentiate the colour of the supernatant solution for different concentrations and to detect the correct status. This problem was overcome by making a modification of Method-2 in Method-3.

Method-3

The results of soil analysis obtained in Method-3 have been presented in Table-7. Like Method-1 and Method-2, Method-3 also worked nicely and gave developed under to Method-1 and Method-2. In this method, the turbidity developed under alkaline condition was made stable by adding HCl. Under this condition, the turbidity developed for two close concentrations became more distinct,

and thus became easier to detect the status of SO_4^{2-} of unknown solution.

Method for rapid determination of soil calcium

The soil samples that used for determination of available sulphur were also extracted with Morgan solution and NH_4OAc solution for the determination of calcium. The extractable Ca were analysed by flame photometer and by proposed kit method for qualitative status (Tables 8 and 9). In general, Morgan solution extracted higher amount of calcium from all soils than NH_4OAc did, however the trend of extractions for different soils remained same. BARC (1997) has classified the calcium status of soil as <300 ppm very low, 301-

600 ppm low, 601-900 ppm medium, 901-1200 ppm optimum, 1201-1500 ppm high and > 1500 ppm very high. According to this grading, the amount of calcium extracted from different soils by NH_4OAc varied from low to very high, where most of the soils fall under optimum level. The same soils were also extracted by Morgan solution and the concentration of calcium in the extract was detected by flame photometer and a portion of the same extract was also tested by the proposed method for inclusion in kit. The results obtained from flame photometric reading as well as the result obtained from testing by kit procedure has been presented in Tables 8 and 9.

Table 6
Concentration and status of calcium in soil extracted by Morgan solution.

Soil Series	Status of Ca by proposed method	Conc. of Ca by standard method (ppm)	Status of Ca according to BARC (1997)
Sonatola	Medium	950	Optimum
Barisal	Medium	900	Medium
Chandra	Medium	710	Medium
Dumuria	Very high	4850	Very high
Ghatail	High	1220	High
Lauta	High	1240	High
Lockdeo	High	1290	High
Nijhuri	High	1210	High
Taherpur	Optimum	1020	Optimum
Tippera	Optimum	970	Optimum

Table 7
Concentration and status of calcium in soil extracted by NH_4OAc solution.

Soil Series	Status of Ca by proposed method	Conc. of Ca by standard method (ppm)	Status of Ca according to BARC (1997)
Sonatola	Low	650	Medium
Barisal	Medium	660	Medium
Chandra	Low	410	Low
Dumuria	Very high	3700	Very high
Ghatail	Optimum	1070	Optimum
Lauta	Medium	940	Optimum
Lockdeo	Optimum	1150	Optimum
Nijhuri	Optimum	1100	Optimum
Taherpur	Optimum	930	Optimum
Tippera	Medium	930	Medium

It appears from Table 8 that the calcium status of soils extracted by Morgan solution falls within the range, medium to very high, where unlike the NH_4OAc extract one step higher results were

obtained in all soils. As evidence, the NH_4OAc extractable calcium of Ghatail, Lauta, Lockdeo and Nijhuri soils may be considered for comparison. The NH_4OAc extractable calcium

status of these soils was of optimum grade, while Morgan solution extractable calcium was of high grade in same soils. Similarly, the status Tippera and Chandra were medium and low for NH_4OAc

whereas for Morgan solution it was optimum and medium, respectively.

Table 8
Status of calcium in soil extracted by Morgan solution under different shaking time (Soil Testing Kit).

Soil Series	Calcium Status				
	1 min. shaking	2 min. shaking	3 min. shaking	4 min. shaking	5 min. shaking
Sonatola	Medium	Medium	Medium	Medium	Medium
Barisal	Medium	Medium	Medium	Medium	Medium
Chandra	Medium	Medium	Medium	Medium	Medium
Dumuria	Very high	Very high	Very high	Very high	Very high
Ghatail	High	High	High	High	High
Lauta	High	High	High	High	High
Lockdeo	High	High	High	High	High
Nijhuri	High	High	High	High	High
Taherpur	Optimum	Optimum	Optimum	Optimum	Optimum
Tippera	Optimum	Optimum	Optimum	Optimum	Optimum

Table 9
Concentration of calcium in soil extract (Morgan solution) under different shaking time (Flame photometry).

Soil Series	Calcium Status				
	1 min. shaking	2 min. shaking	3 min. shaking	4 min. shaking	5 min. shaking
Sonatola	950	950	950	940	940
Barisal	890	900	900	900	900
Chandra	700	700	710	700	710
Dumuria	4900	4900	4850	4800	4900
Ghatail	1220	1220	1230	1230	1230
Lauta	1240	1240	1240	1250	1240
Lockdeo	1290	1280	1280	1270	1280
Nijhuri	1200	1200	1210	1190	1200
Taherpur	1020	1030	1220	1040	1020
Tippera	960	970	960	970	960

Shaking time is often considered as an important factor in getting higher/lower results of a nutrient element. The farmers also may not be able strictly maintain the shaking time during analysis. In order to see the effect of shaking time on the amount of extractable calcium, another test was performed maintaining five different shaking times for all of these soils under study. The results of this test are presented in Tables 10 and 11. The numerical values were compared with BARC (1997) grading and found medium to very high. The test of the same extract by Kit procedure also indicated the status medium to high keeping a very good relation with BARC (1997) grading. This also

indicated the suitability of the proposed method for testing the available calcium status of different soils.

The conventional method for analysis S and Ca need very costly equipment and technical personnel for operation and maintenance in addition to chemicals. The methods are also complex and time consuming. All of these make the cost of analysis very high. On the other hand, in these proposed rapid methods a common extracting reagent is needed for both S and Ca and a colour/turbidity developing reagent for each element besides the colour chart. In view of above

these proposed methods for S and Ca were more convenient and economic.

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