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RESEARCH ARTICLE

SOLUBILITY ENHANCEMENT: A POTENT TOOL FOR GYPSUM DOSE REDUCTION IN SODIC SOIL RECLAMATION

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ABSTRACT

Gypsum which provides readily available source of Ca^{2+} ions to replace Na^+ ions from the cation exchange complex of soil has been used as a common chemical amendment in the reclamation of sodic soil. As a non-renewable resource in nature, its dwindling scenario has now become imperative to find either an alternate sources of gypsum or to increase the efficacy of gypsum to reduce its dose in the reclamation of such sodic soils. The present study was undertaken with the aim to enhance the solubility of gypsum using different chemical formulations both in laboratory and pot culture experiment. It was observed that the addition of KNO_3, NH_4NO_3 and NaCl to gypsum, increased the Ca^{2+} ion to 39.73 %, 60.59 % and 68.05 %, respectively. The maximum increase in the Ca^{2+} content was noticed in gypsum saturated solution containing NH_4NO_3 + citric acid and NaCl + citric acid combination which was 94.53 % and 91.22 %, respectively. The increase of Ca^{2+} ion due to the increased dissolution of gypsum resulted in saving of 75% and 50% of its doses in the reclamation of sodic soil when compared with the quantity required under normal existing reclamation process (50 GR).

INTRODUCTION

The land for agriculture is constantly shrinking due to the rapid rise in the population and urbanization. Therefore, to meet challenges of global food security of the people, it is now imperative to find ways and means to improve the waste or degraded lands to harness their productive potential. The salt affected soils also come under these categories and are treated as an important ecological entity in the landscape of any arid and semi-arid regions**.** As per the estimate (Qadir *et al*., 2006), at least 20 per cent of the world's irrigated land is salt-affected and about 60 per cent of salt-affected soils are sodic and saline-sodic in nature (Tanji, 1990). India is also confronting the problems of soil salinity and sodicity (Gupta and Abrol, 2000). The sodic soils are characterized by excess level of sodium ion (Na^+) in the soil solution phase as well as on cation exchange complex. As a result, it exhibit unique structural problems as a result of certain physical processes (slaking, swelling, and dispersion of clay) and specific conditions (surfacing, crusting and hardsetting) (Summer, 1993; Qadir and Schubert, 2002). These problems may reduce infiltration rate, air movement, plant available water holding capacity, root penetration, seedling emergence and increase runoff and erosion (Oster and Jayawardane, 1998) and ultimately leads to decreased productivity. These soils are having pHs > 8.5, exchangeable sodium percentage (ESP) >15 and varying electrical conductivity (EC). In an approximation to ESP, sodium adsorption ratio (SAR) values have also been widely used and a value (SAR=13) is taken as threshold above which the soil becomes dispersive when water is applied (Qadir *et al.,* 2006). These soils have been ameliorated so far by providing a readily available source of calcium (Ca^{2+}) that replaces excess Na^+ on the cation exchange complex. Mineral gypsum ($CaSO_4.2H_2O$) has been used extensively so far as chemical amendment in the reclamation of sodic soils (Gobran *et al.*, 1982) which provides readily available source of Ca^{2+} ions and is found around the world in sedimentary rock formation from where it is mined or quarried. Currently, for the reclamation of sodic soil (0-15 cm depth) the gypsum requirement (GR) for a soil with pH ~ 10.0, 15 t/ha is recommended which is equivalent to 50 GR. Since mineral gypsum is a non-renewable resource and dwindling day by day due to its exhaustive use both in agriculture as well as in industries, it has now become imperative to find either an alternate sources of gypsum or to devise some tool to increase the efficacy of gypsum and reducing the gypsum doses in the reclamation of such sodic soils. Gypsum is moderately water soluble having solubility value of 0.2 to 0.25 % only at 25° C (Bock, 1961). Therefore, by increasing the aqueous solubility of gypsum, the efficacy of gypsum in the reclamation of sodic soils could be increased and thereby reducing the gypsum doses. The current research aimed to increase the solubility of gypsum for reducing gypsum doses and to evaluate the change in soil properties after the reclamation of sodic soil that is responsible for soil sodicity.

MATERIALS AND METHOD

Theoretical

Whenever a sparingly soluble substance like gypsum ($CaSO_4.2H_2O$) is placed in water, the solvent uses ion-dipole forces to disengage the ions from the lattice positions and the process continues till equilibrium is reached i.e. the rate of dissolution of substance equals the rate of precipitation from the saturated solution. The solubility of gypsum in water or aqueous medium can be described by equilibrium reaction given by Lindsay (1979) and may be represented as:

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$CaSO_4.2H_2O\ (gypsum) \leftrightarrow Ca^{2+} + SO_4^{2-} + 2\,H_2O,\ \log K^0 = -\,4.64$

Where K^o is the equilibrium constant of the reaction and is equal to:

$$K^o = \frac{a_{Ca^{2+}}\, a_{SO_4^{2-}}\, a_{H_2O^2}}{a_{gypsum}}$$

Since the activity of pure solid and water is unity at equilibrium, the above equation reduced to $K^o_{sp} = a_{Ca^{2+}}\, a_{SO_4^{2-}}$

$= mCa^{2+}\ \gamma\ Ca^{2+}\ mSO_4^{2-}\ \gamma\ SO_4^{2-}$

Where K^o_{sp} is the solubility product constant for $CaSO_4.2H_2O$ at zero ionic strength, $a_{Ca^{2+}}$ and $a_{SO_4^{2-}}$ are the activities of ions, mCa^{2+} and mSO_4^{2-} are the molarities of the ions and $\gamma\ Ca^{2+}$ and $\gamma\ SO_4^{2-}$ are their respective activity coefficients.

However, the solubility of ionic compound increases when the ionic strength of the solution increases due to the presence of other electrolyte from the substance containing no common ion with the salt i.e. $S_O = \frac{\sqrt{K_{sp}}}{\gamma_\pm}$, where mean ionic activity coefficient $\gamma_\pm \neq 1$ because of the change in the ionic strength of solution and S_o is the solubility of salt. The mean ionic activity coefficient can be calculated using Debye Huckel limiting law:

$\log\gamma_\pm = -\,0.511\langle Z_\pm\, Z_-\rangle\sqrt{I}$, where Z_+ and Z_- are the charge number of cations and anions, respectively and I is the ionic strength of the solution. The ionic strength is a measure of electrical environment in solution and is defined as:

$\mu = (\frac{1}{2}\sum C_i Z_i^2)$ where μ is the ionic strength of the solution; Ci (C1, C2, C3………) is the concentration of various ions in g ionic wt. per litre while Zi (Z1, Z2, Z3……..) are the valencies of the respective ions. The activity of each ion is written as the product of its activity coefficient and concentration.

i.e. $a_+ = \gamma_+ m_+ ;\ \gamma_+ = a_+/m_+$

lly $a_- = \gamma_- m_-$ and $\gamma_- = a_-/\,m_-$

Experimental

Laboratory Experiment

Experiment 1

5 g $CaSO_4.2H_2O$ (GR grade Merck) taken in a 250 ml Erlenmeyer conical flask and added with 100 ml of distilled water. The material was shaken for 1, 2, 4, 6, 8, 10, 12, 16, 20 and 24 hrs in a horizontal rotary shaker in the laboratory. The contents were filtered and the calcium was determined using versenate method (Richards, 1954) in the extract collected.

Experiment 2

5 g $CaSO_4.2H_2O$ (GR grade Merck) taken in a 250 ml Erlenmeyer conical flask and were added with chemicals as illustrated below in various formulations:

F1 -5 g $CaSO_4.2H_2O$ + 100 ml distilled water
F2 - 5 g $CaSO_4.2H_2O$ + 1 g citric acid +100 ml distilled water
F3 - 5 g $CaSO_4.2H_2O$ + 1 g KNO_3 +100 ml distilled water
F4 - 5 g $CaSO_4.2H_2O$ + 1 g KNO_3 +1 g citric acid +100 ml distilled water
F5 - 5 g $CaSO_4.2H_2O$ + 1 g NH_4NO_3 +100 ml distilled water
F6 - 5 g $CaSO_4.2H_2O$ + 1 g NH_4NO_3 +1 g citric acid + 100 ml distilled water
F7 - 5 g $CaSO_4.2H_2O$ + 1 g NaCl + 100 ml distilled water
F8 - 5 g $CaSO_4.2H_2O$ + 1 g NaCl +1 g citric acid + 100 ml distilled water

The above contents were shaken in horizontal rotary shaker for 16 hr and Ca content was determined in the extract.

Experiment 3

20 g sodic soil (pH 10.34) taken in a 100 ml beaker and added with 40 ml of distilled water whereas in the other beakers 20 g sodic soil (pH 10.34) is shaken by a glass rod with 40 ml extracts obtained in experiment 2 and kept for ½ hour. After ½ hour pH and EC were measured potentiometrically using Orion ion analyzer with the help of pH electrode and conductivity electrode, respectively.

Pot culture experiment

Based on the observations in the laboratory experiments, a pot culture experiment was conducted in rice and wheat during kharif and rabi season, respectively in the same pots. The sodic soil (10 Kgs) was taken in each pot and the experiment was carried out in 3 replicates under various treatments:

T1= 50 GR @ 15 t/ha Gypsum (75 g)
T2 = 25 GR @ 7.5 t/ha Gypsum (37.5 g)
T3= 12.5 GR @ 3.75 t/ha Gypsum (18.75 g)
T4= 25 GR @ 7.5 t/ha Gypsum (37.5 g) + 2 g KNO_3
T5= 12.5 GR @ 3.75 t/ha Gypsum (18.75 g) + 1 g KNO_3 + 1 g Citric acid
T6= 25 GR @ 7.5 t/ha Gypsum (37.5 g) + 2 g NH_4NO_3
T7= 12.5 GR @ 3.75 t/ha Gypsum (18.75 g) + 1 g NH_4NO_3 + 1 g Citric acid
T8= 25 GR @ 7.5 t/ha Gypsum (37.5 g) + 2 g NaCl
T9=12.5 GR @ 3.75 t/ha Gypsum (18.75 g) + 2 g NaCl + 1 g Citric acid

Rice (cultivar Moti) seedlings were transplanted in all the pots. A basal application of half of total N *i.e.* @150 kg N/ha and full dose of P @ 60 kg P_2O_5/ha and 40 kg K_2O/ha was applied at the time of transplanting. Rest amount of N was applied in two equal splits at the time of tillering and panicle initiation. The irrigation water (5 cm) ponding was maintained in all treatments. On the same pots, wheat was taken in rabi season. The cultivar KRL19 was taken and the fertilizer (N: P_2O_5 : K_2O = 60 : 60 : 40 kg ha^{-1}) was applied as basal dose to all pots. Again half of total N, i.e. 60 kg N ha^{-1}, was applied in two equal splits at the time of first and second irrigations, respectively. At the harvest of wheat crop, the soil samples from each pot were taken, air dried, ground and sieved through 2 mm sieve. The soil parameters pH, EC, CO_3, HCO_3, Cl, Ca, Mg, Na and K were determined. Also SAR in various samples were calculated using equation:

$$SAR = \frac{Na}{\sqrt{\frac{(Ca+Mg)}{2}}}$$

Statistical analysis

Treatment means were compared using ANOVA and the level of significance considered was $p > 0.01$ (Gomez and Gomez 1984).

RESULTS

The initial characteristics of experimental soil which is presented in Table 1 clearly indicated that the soil is highly sodic in nature with respect to pH and SAR values. The EC was also less than 4 dS m^{-1}. In the experiment I, shaking of gypsum solution for 1 hr resulted in only 28.5 me/l of Ca^{2+} content and a value of 30.2 meq/l was found to be the highest both at 16 hrs and 24 hrs shaking (Fig. 1). The highest value was taken for the comparison. In the experiment II, the addition of inorganic salts potassium nitrate (KNO_3) ammonium nitrate (NH_4NO_3) and sodium chloride (NaCl) significantly increased the Ca^{2+} ion in gypsum saturated solution. Due to the addition of KNO_3, NH_4NO_3 and NaCl to the gypsum, the percent increase in the Ca^{2+} ion was 39.73 %, 60.59 % and 68.05 %, respectively with respect to formulation F1 (Table 2). The maximum increase in the Ca^{2+} content was noticed in gypsum saturated solution containing NH_4NO_3 + citric acid (F6) and NaCl + Citric acid combination (F8) which was 94.53 % and 91.22 %, respectively. The pH and EC of soil taken in the 1:2 soil suspension in the extracts (F1 to F8), showed a remarkable decreased value of pH (8.7, 7.86 and 8.74) in the extract of KNO_3, NH_4NO_3 and NaCl (Table 3). The decreased pH values were also observed in the formulations (F2, F4, F6 and F8) containing citric acid. The pH measurement of the soil (1:2 suspensions) after the wheat harvest indicated a significant decrease in comparison to pH of soil before rice plantation (after gypsum application) in pot experiment (Fig. 2). With regard to pH of the soil extract (pHe) (Table 4), the treatment T4 and T6 was found to be at par with each other whereas treatments T8 and T9 performed better even compared to T1 (50 GR gypsum). The SAR values in all the treatments except control were found to be low (SAR< 13). A decrease of CO_3+HCO_3 value was also found in all the treatments as compared to the control.

Table 1. Initial characteristics of experimental soil

pHe	10.34
ECe, dS^{-1}	2.76
CO_3, mel^{-1}	2.5
HCO_3, mel^{-1}	19.6
Cl, mel^{-1}	5.5
SO_4^{2-}, mel^{-1}	0
Ca, mel^{-1}	0.5
Mg, mel^{-1}	1.1
Na, mel^{-1}	25.6
K, mel^{-1}	0.01
SAR, cmol kg^{-1}	28.62

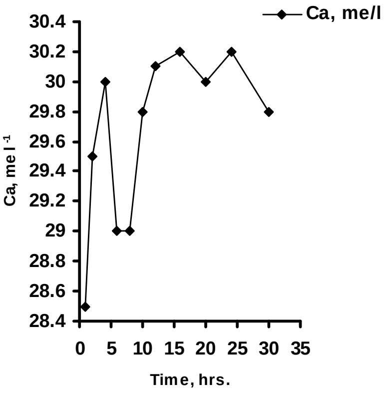

Fig.1. Variation of Calcium content with time in saturated gypsum solution

Table 2. Calcium content under different formulations

Formulations	Ca, me/l	% increase
F1	30.2	-
F2	38.5	27.48
F3	42.5	40.73
F4	46	52.32
F5	48.5	60.59
F6	58.8	94.7
F7	50.9	68.05
F8	57.8	91.39

Table 3. The pH and EC of sodic soil in the extract of different formulation

Formulations	$pH_{1:2}$	$EC_{1:2}$, dS m^{-1}
F1	9.57	0.72
F2	3.78	4.81
F3	8.70	1.27
F4	3.65	4.60
F5	7.86	15.20
F6	2.35	15.74
F7	8.74	18.03
F8	3.50	18.97
Control	0.35	1.36

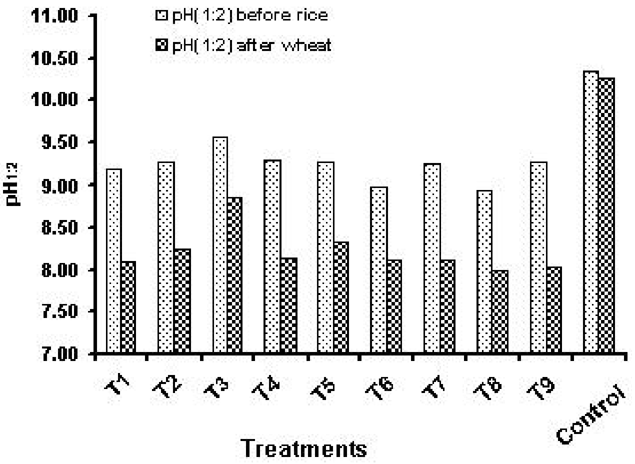

Fig. 2. pH variation in 1:2 soil suspension in different treatments

DISCUSSION

The amount of Ca^{2+} content (30.2 meq/l) in experiment I was found to be higher than the theoretical value of 29.1 meq/l or 582 mg Ca^{2+} ion/l in gypsum saturated solution (Elrashidi *et al.*, 2007). Even, in the determination of gypsum requirement by Schoonover (1962), the saturated gypsum solution contained at least 28 meq/l Ca^{2+} ion due to low solubility in aqueous solution. The addition of salts in the formulations (F2 to F8) increased the solubility of gypsum. This could be attributed to the fact that the solubility increases when an electrolyte with no ions in common is added to the sparingly soluble salt. This effect is known as *Salt Effect* which depends upon the valency of the ions added to the solution. The addition of a salt with no common ion will increase the ionic strength $(\frac{1}{2}\sum C_i Z_i^2)$ of the solution resulting in the lowering of the activity coefficients of the ions furnished by the sparingly soluble salts. The hydration reaction has been shown to be accelerated when other salts such as NaCl are

Table 4. The chemical properties of soil after wheat harvest

Treatments	pHe	ECe, dS m^{-1}	CO_3	HCO_3	Cl	SO_4	Ca	Mg	Na	K	SAR, cmole kg^{-1}
			------------	------------	------------	me/l	------------	------------	------------		
T1	8.31	0.53	1	2.5	1.5	1	2	1	2.55	0.07	2.55
T2	8.75	0.74	1.5	2.5	2	1.5	1.5	0.5	5.34	0.09	7.55
T3	8.81	0.79	1	2.5	3	1.3	1	0.5	6.28	0.06	8.88
T4	8.18	0.53	0.5	2.5	1.5	1	2	0.5	3.06	0.09	4.33
T5	8.57	0.57	1	2.5	1	1.4	1	0.5	4.05	0.11	5.73
T6	8.21	0.50	0.5	1.5	2	1.1	1.5	1.5	1.92	0.13	1.57
T7	8.36	0.55	1	2.5	1.5	1	2	1	2.96	0.09	2.96
T8	8.00	0.41	0	1.5	2.5	0.5	2	0.5	1.83	0.11	2.59
T9	7.98	0.43	0	1	2.5	1	2	0.5	2.03	0.12	2.87
Control	10.07	2.71	2	18.9	5.1	1.2	0.5	1.5	25	0.01	25.00

present (Charola *et al.,* 2007). The dissolution-exchange reactions though may appear simple, involves simultaneous mineral dissolution, cation exchange and solute movement (Gupta and Abrol, 1990). The addition of unlike ions like KNO_3, NH_4NO_3 increased the gypsum solubility and is in conformity with the findings of earlier findings of Charola *et al.* (2007), according to which the aqueous solubility of gypsum increased significantly by the addition of $NaNO_3$, KNO_3 and $Mg(NO_3)_2$. Moreover, another reason for the higher dissolution rate of gypsum may be ascribed to the increased order of reaction due to the addition of KNO_3, NH_4NO_3. This is because the higher order of a reaction leads to increased rate of reaction (Laidler, 1965). Citric acid is a weak organic acid, soluble in water and is an effective chelator of metals, the addition of which increased the acidity and led to the protonation of sulphate ion of gypsum toform bisulphate:

$H^+ + SO_4^{2-} \longrightarrow HSO_4^-$. This decreased the sulphate concentration and drives further dissolution of gypsum (Wolfe and Bennett, 2011). Gypsum is one of the most commonly occurring sulphate mineral in soil which may be present in trace amounts in soils or dominate the pedon as in gypsiferous soil with a gypsic or petrogypsic horizon (Eswaran and Zi-Tong, 1991). The relatively low solubility of gypsum in aqueous systems suggests that it may be present as crystalline material in water-saturated soil paste (Lagerwerff *et al*., 1965). However, dissolution increases with SAR, ionic strength, ion-pair association, and C_{Mg}/C_{Ca} ratio (Tanji, 1969; Oster and Rhoades, 1975; Oster, 1982). The dissolution rate of gypsum is also controlled by film diffusion and is a function of the difference between the solution concentration at saturation and solution concentration at a particular time (Kemper *et al*., 1975). In the pot experiment, the reason for the treatments T8 and T9 to be better even with T1 (50 GR gypsum) is obviously due to i) the increased solubility of gypsum which may be due to the release of NH_4^+ and K^+ ion from NH_4NO_3 and KNO_3, respectively and gets exchanged with the Na^+ ion from the soil exchange sites. This in turn reacts with SO_4^{2-} ion to form soluble Na_2SO_4, which might trigger the dissociation of gypsum further ii) the release of H^+ ion from citric acid also exchanges Na^+ ions from the exchangeable sites, as H^+ ion is placed higher in lyotropic series. The order is: $Al^{3+}>H^+>Ca^{2+}>Mg^{2+}>K^+\approx NH_4^+>Na^+$. Both the reasons might have resulted in the reduction of ESP and improvement of soil structure.

Another possible reason is that Na^+ ions released from the soil exchange sites combine with the citrate ions to form a hydrotropic agent, sodium citrate. The term hydrotropic agent was first coined by Neuberg (1916) to designate anionic organic salts which at high concentration increases the aqueous solubility of poorly soluble solute (Neuberg, 1916). The hydrotropy is one of the solubility enhancement techniques which increases solubility to many folds with the use of hydrotropes like sodium citrate, sodium benzoate, urea etc and has wide application in the chemical industries for the preparation of aqueous dye solution and in drug industries (Jain *et al*. 2010, Kapadia *et al.* 2011). This solubilization process involves cooperative intermolecular interaction with several balancing molecular forces rather than simply a specific complexation events or miscibility, micellar solubilization, cosolvency and salting in (Jain *et al.,* 2010) and consists of both hydrophilic and hydrophobic part. In soil system, dissolution and precipitation of gypsum occurs simultaneously till it attain equilibrium, which is governed by the various conditions. If ionic activity product (IAP) of gypsum exceeds solubility product (K_{sp}) then precipitation occurs but when it is less than $K_{sp,}$ dissolution occurs. The solution will be in equilibrium with the solid when both IAP and K_{sp} will be equal. The precipitation/dissolution of gypsum has been described by Suarez and Simunek (1996). In present case, multiple salts are in solution and their interactions have influenced solubility. In the treatment (T8 and T9), the solubility might have increased due to the presence of NaCl which is in agreement with earlier researcher (Shaw *et al*., 1987) stated that gypsum solubility is potentially three times more in presence of NaCl. Secondly, the formation of a hydrotropic agent due to the presence of citric acid in treatment T9 might have increased the solubility of gypsum further. These hydrotropic agents are the molecules having a planar hydrophobic structure brought into solution by a polar group. The increase of Ca^{2+} ion due to the increased dissolution of gypsum resulted a saving of 75% gypsum and 50% gypsum in the reclamation of sodic soil when compared with the quantity required in the treatment with 50 GR and 25 GR mineral gypsum, respectively.

Conclusion

The study clearly indicated an increase of 27.48 to 94.7 % in Ca^{2+} content in various formulations. This increase led a saving of 75% gypsum in the reclamation of sodic soil in pot experiment when compared with the quantity required in the treatment with 50 GR mineral gypsum for reclamation. In view of dwindling scenario of mineral gypsum, the tool of solubility enhancement may be used in increasing the efficacy of gypsum by reducing its doses in the reclamation of sodic soils. This study emphasizes the importance of further experimentation under field conditions to develop a cost effective and eco-friendly sodic soil reclamation programme.

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