

## SYNTHESIS AND CHARACTERISATION OF NANO-GYPSUM FOR EFFECTIVE REMEDIATION OF SODIC SOIL OF TAMIL NADU, INDIA

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**Introduction:** A green hood use experiment was conducted during 2011-12 in the Department of Soil Science and Agricultural Chemistry, Tamil Nadu Agricultural University, Coimbatore to evaluate the efficacy of nano-gypsum ((NG) in the reclamation of sodic soil in comparison with conventional gypsum (CG) at four levels of Gypsum Requirement (GR) @ 25, 50, 75 and 100 % and control, replicated thrice. The sodic soil collected from AnbilDharmalingam Agricultural College and Research Institute (ADAC &RI), NavalurKuttapattu, Tiruchirappalli district was non calcareous, non salinesodic of Panjappur series-Typicustropept with a pH of 9.38 and ESP of 44.07. Nano-gypsum was synthesized and characterized in the Department of Nano Science and Technology, TNAU, Coimbatore and observed to be clustered and consolidated with a surface area of  $900\text{m}^2$  and the Raman and FT-IR spectra confirmed the presence of gypsum loaded into the nano-clay and the XRD pattern depicted the reduction in d-spacing that coincide with the ion attachment between the lattices.

The results of the percolation study conducted in the percolation reactor revealed the significant displacement of  $\text{Na}^+$  from the exchangeable clay complex by the  $\text{Ca}^{2+}$  of nano-gypsum as indicated by the declining concentration of  $\text{Ca}^{2+}$  in the leachate. The results of the green house experiment revealed that the pH of the sodic soil significantly reduced to 7.43 and ESP to 9.38 by the treatment with 100 % GR as nano-gypsum confirming the reclamation of the sodic soil. A significant increase in the exchangeable  $\text{Ca}^{2+}$  in the clay complex and promiscuous decline in exchangeable  $\text{Na}^+$  and higher concentration of  $\text{Na}^+$  in the leachate recorded by the application of 100 % GR as nano-gypsum indicates the successful reclamation of sodic soil. The significant leaching of anions viz.,  $\text{HCO}_3^-$  and  $\text{SO}_4^{2-}$  was recorded. Reclamation with nano-gypsum @ 100 % GR improved the physical properties of the sodic soil by reducing the bulk density to  $1.26\text{ Mg m}^{-3}$  from  $1.31\text{ Mg m}^{-3}$  in the control, increasing the porosity by 6 % and improving the hydraulic conductivity to  $0.52\text{ cm hr}^{-1}$  from  $0.43\text{ cm hr}^{-1}$ .

**Keywords:** FTIR, Nano clay, Nano -gypsum, Sodic soil, XRD, SEM.

**Introduction:** Soil degradation caused by salinization and sodification is one of the greatest concerns associated with crop yield reductions in the country vis-à-vis world. Of the various problem soils in the world, saline and sodic soils occupy an extent of 323 million hectares (FAO, 2009). In India, out of 329 million hectares of total geographical area, the arid and semi-arid occupy more than one third of the area (127.4 m ha). The salt affected soils occurring in these zones occupy 12 m ha spread over 15 states of the country. These salt affected soil zones comprise of 4.12 m ha of alkali soil, 3.26 m ha of saline soil and 4.62 m ha of saline alkali soils. Among these salt affected soils, alkali soils are found to be highly problematic for crop production because of very poor physical and chemical environment particularly in irrigated areas. Sodicity problem in irrigated agriculture is becoming more and more serious because of poor quality water, lack of adequate knowledge about soils and poor management practices. The amelioration of these alkali soils is not only expensive but also time consuming and laborious (Gupta *et al.*, 1995).

The typical sodic soils are characterized by high pH (>8.5), excess of exchangeable sodium (ESP>15), lower EC (<4.0) and possess predominant salts viz. sodium

bicarbonate, sodium carbonate and sodium silicate, that are capable of alkaline hydrolysis (Pagaria *et al.*, 2011) besides adverse soil physical properties. In sodic soils, the dominant cation on the exchange complex is sodium, which disperses clay and imparts adverse soil physical conditions including poor soil-water behaviour to these soils. As a result, these soils have poor water permeability (Singh *et al.*, 2009).

Reclamation of alkali soils is essential to improve the productivity of the soil and to gain advantage of inherent fertility. The problem soils can successfully be cultivated by removing excessive soluble salts and exchangeable sodium through reclamation techniques. Various amendments like gypsum, sulfur and acids may be used for amelioration of these soils (Biggar, 1996). During the reclamation process, Na in the exchangeable clay complex is replaced by Ca derived from the gypsum, a commonly used amendment. Because of low solubility of gypsum and calcareous nature of soils, its efficiency is reduced. Efforts are being made to bring these soils under cultivation through application of amendments, preferably gypsum, and adoption of rice (*Oryza sativa* L.) based cropping sequence (Swarup and Singh, 1989). Traditionally, gypsum is recommended for the reclamation of sodic soils but ability to reclaim the

soil depends on the quality (fineness and solubility) and quantity of gypsum used. With an idea to explore the possibilities of nanotechnology in soil reclamation and to improve the efficiency and economise the gypsum requirement, a maiden attempt was made to design nano- gypsum for soil reclamation. Nanotechnology deals with small particles with the dimension of 1-100 nm (one billionth of a meter). These particles have high surface mass ratio and are capable of improving the agricultural inputs including gypsum. Nanotechnology applications in agriculture are gradually transforming the theoretical possibilities into the practical applications. The potential is increasing with suitable techniques and sensors being identified for precision agriculture, natural resource management, early detection of pathogens and contaminants in food products, efficient delivery systems for agrochemicals such as fertilizers and pesticides, improved systems integration for food processing, packaging and other areas like monitoring agricultural and food system security (Chauet *al.*, 2007; Subramanian and Tarafdar, 2011). The role of nanotechnology in soil remediation/reclamation is yet to be attempted.

The present research work was aimed to synthesize nano-gypsum locally and to study the effect of nano-gypsum on the reclamation of sodic soil of Tamil Nadu, India.

**Materials And Methods:** The present investigation was carried out with the primary objective of assessing the efficiency of nano-gypsum in reclamation of sodic soils. The nano-gypsum was synthesized, characterized and compared with the conventional gypsum at different levels to study its efficacy in the reclamation of sodic soils.

**Estimation of Gypsum Requirement (GR) for Sodic soil:** The soil samples for the experiment was collected from AC&RI, Navalurkuttapattu, Trichy, Tamil Nadu, India to evaluate the efficacy of the synthesised nano-gypsum and to study the relative performance with conventional gypsum. The initial soil was analysed for the physico chemical properties and the GR was calculated (Table 1). The experimental soil was assessed for sodicity by measuring the soil reaction (Jackson, 1973). Subsequently the Exchangeable Sodium was estimated (Black, 1965) and Exchangeable Sodium Percentage (ESP) of the soil was calculated. The gypsum requirement (GR) was calculated based on the ESP of the Soil (Das, 1996). The quantity of gypsum required to bring down the ESP to a desired level of ten (10) was calculated as per the following formula. **Gypsum requirement(GR)**

$$\text{(me of Ca}^{2+}\text{/100 g soil)} = \frac{\text{ESP(initial)-ESP(final)} \times \text{CEC}}{100}$$

The calculated quantity of gypsum was applied through two sources viz., conventional gypsum and nano- gypsum.

#### **Synthesis of nano-gypsum**

**Source:** The nano-clay (montmorillonite) was purchased from M/s Sigma Adrich Chemicals, Bangalore. The montmorillonitenano-clay had a dimension of 30-120 nm in size and the analar grade of gypsum was used for fortification

**Preparation of nano-gypsum:** The required quantity of gypsum as per the gypsum requirement (GR) was fortified into the substrate (nano-clay) to achieve the nano-gypsum. Accurately 500 g of analar grade gypsum was dissolved in 2000 ml of distilled water and the solution was transferred to 500 g nano-clay. The content was homogenised in an ultra sonicator for 30 minutes and kept undisturbed for 15 minutes. The sonication was repeated for another 30 minutes. The slurry was spread in a metal tray and kept in hot air oven at 70°C for 2 to 3 days until the material reached state of dry powder.

The dried sample of nano-gypsum was pulverised in a high energy ball mill and nearly one kilogram of nano-gypsum was produced.

#### **Characterization of nano-clay and nano-gypsum Particle Size Analysis:**

The nano-clay (montmorillonite) sample was tested for particle size analysis. Particle Size Analysis is an analytical technique by which the distribution of sizes in a sample of particulate material can be measured. Particle Size Analyzer (Malvern, Zetasizer Ver.6.01) was used to measure the dimension of the nano-clay fortified with or without gypsum. Accurately, 100 mg of nano-clay sample was dispersed in 20 ml of acetone and sonicated using ultrasonic processor for twenty minutes at 250 rpm and the size distribution of the particle and average diameter of the nano particles were measured in a particle size analyzer.

**Scanning Electron Microscopy (SEM):** The Scanning Electron Microscope (SEM) uses a focused beam of high-energy electrons to generate a variety of signals at the surface of solid specimens. The signals that derive from electron-sample interactions reveal information about the sample including external morphology (texture), chemical composition, and crystalline structure and orientation of materials making up the sample. Areas ranging from approximately 1 cm to 5 microns in width can be imaged in a scanning mode using conventional SEM

techniques (magnification ranging from 20X to approximately 30,000X, spatial resolution of 50 to 100 nm).

Nano-clay loaded with gypsum or in composite formulation was placed in the specimen chamber and mounted rigidly on a specimen holder called a specimen stub. The SEM used for the analysis was FEI, Quanta 200, Netherland, for taking images of nano-clay samples. About 50 to 100 mg of nano-clay sample was dusted on the carbon conducting tape. Then the tape was mounted on sample stage and the images were taken in 16,000 magnification and 12.50KV.

#### Energy Dispersive X-ray Spectroscopy (EDAX):

Energy Dispersive X-Ray Spectroscopy (EDS or EDX) is a chemical microanalysis technique used in conjunction with scanning electron microscopy (SEM). The EDS technique detects X-rays emitted from the sample during bombardment by an electron beam to characterize the elemental composition of the analyzed volume. Features or phases as small as 1  $\mu\text{m}$  or less can be analyzed. The quantitative analysis of nano-clay samples was done by FEI Quanta 200 EDAX. About 100 - 200 mg of nano-clay sample was dusted on the carbon conducting tape.

**Raman Spectroscopy:** Raman spectroscopy is a spectroscopic technique based on inelastic scattering of monochromatic light, usually from a laser source. Raman spectroscopy can be used to study solid, liquid and gaseous samples.

Raman spectrum is a spectral "fingerprint". The Raman spectrum was measured for the nano clay loaded with or without gypsum. Raman spectroscopy used for measurement was Raman spectrum Model-R- 3000- QE. The powder dried sample kept in a polythene bag was spread to an extent of 1  $\text{cm}^2$  and Raman probe was placed on the sample pockets without exposing the sample directly to the probe.

**X-ray Diffraction:** X-ray Diffraction (XRD) is a rapid analytical technique primarily used for surface area determination, phase identification of a crystalline material and can provide information on unit cell dimensions.

Approximately one gram of nano clay loaded with or without gypsum was dusted on a glass substrate. Care was taken that the surface on the glass substrate is even, then the false substrate was mounted on the sample stage and diffraction was measured. From this an automatic strip chart record of the sample peak was obtained. The well known Deby-Scherrer formula provides the relationship between mean crystallite size and diffraction line breadth. For the work reported herein, the following form of the equation was employed:

$$D = \frac{0.9\lambda}{(\beta - \beta_0) \cos \theta}$$

Where,

- D = Crystallite size (nm)
- $\lambda$  = X-ray wavelength 0.15418nm
- $\beta$  = Breadth at half maximum at intensity of a peak representing the compound in question
- $\beta_0$  = Breadth at half maximum at intensity of a highly crystalline material
- $\theta$  = Bragg's angle

#### Fourier Transform Infrared Spectroscopy (FT-IR)

Fourier Transform Infrared Spectroscopy (FTIR) is a technique which is used to obtain an infrared spectrum of absorption, emission, photoconductivity or Raman scattering of a solid, liquid or gas. The nano clay powder was compressed into a thin pellet which can be analyzed

For the preparation of samples, nano-clay loaded with or without gypsum and IR transparent material like KBr was mixed in the ratio of 2:1 in a mortar and pestle for 30 minutes. Then the mixture was converted into pellets by pressing the prepared mixture with a hydraulic or hand press into a hard disk. The pellet, ideally 0.5 to 1 mm thick was then placed in a transmission holder and scanned. Typically, the pellet technique provides good quality spectra with a wide spectral range and no interfering absorbance bands.

#### Percolation study to assess the efficiency of nano-gypsum:

The percolation reactor was designed in order to assess the efficacy of nano-gypsum in a column of soil with a constant flow of solution as fabricated by Hemandezet *al.*, (1994). The percolation reactor consists of a Teflon cylinder (internal diameter of 2.5 cm and height of 15 cm) through the top of which deionized water is continuously pumped at a flow rate of 66 ml per day.

In this technique, a column of test soil was loaded in a sample chamber. About 1 g of nano-gypsum loaded with varying levels of  $\text{CaSO}_4$  was overlaid above the soil column. A consistent moisture regime was maintained by constant flow of water in order to consistently dissolve the nano-gypsum which can percolate constantly through the moist column of soil. The leachate was collected from the 5<sup>th</sup> day for a period of 60 days at an interval of 5 days. The  $\text{Ca}^{2+}$  content was measured in the leachate regularly (Jackson, 1973) to assess the efficiency of nano-gypsum.

#### Physico chemical properties of nano- clay and nano- gypsum

The synthesised nano- gypsum and the nano- clay

used for the study was analysed for the physico chemical properties as per the standard procedures listed in Table 1

Parameter	Methodology	Reference
<b>Physical properties</b> Bulk density, Particle density and Pore space Colour	Core sampler method Munsell colour chart	Gupta and Dakshinamoorthy (1981)
<b>Physico-chemical properties</b> Soil reaction (pH) and Electrical conductivity (EC) Cation exchange capacity	1:2.5 nano clay: water suspension Copper (II) ion and Triethylenetetramine method	Gupta and Dakshinamoorthy (1981) Jackson, 1973 Meier, 1999

**Treatment combinations:** To study the effect of synthesized nano-gypsum on the reclamation of sodic soil, it was compared with conventional gypsum at different levels as follows.

Factors	Two i. Source of amendment (A) and ii. Levels (L)
Sources	i. Nano-gypsum (NG) ii. Conventional gypsum (CG)
Levels	(i) Control (0%GR), (ii) 25%GR, (iii) 50%GR, (iv) 75%GR and (v) 100%GR.

The treatments were imposed as per the following combinations.

A1L1	Control	A2L1	Control
A1L2	25 % GR as nano-gypsum	A2L2	25 % GR as conventional gypsum
A1L3	50 % GR as nano-gypsum	A2L3	50 % GR as conventional gypsum
A1L4	75 % GR as nano-gypsum	A2L4	75 % GR as conventional gypsum
A1L5	100 % GR as nano-gypsum	A2L5	100 % GR as conventional gypsum

### Results And Discussion:

**Physico chemical properties of the experimental sodic soil (Table 2):** The sodic soil for the experiment was collected and transported from the fields of the Agricultural College and Research Institute, Navalurkuttapattu, Tiruchirappalli district, (erstwhile Soil Salinity Research Centre) the component college of TNAU, Tamil Nadu, India. The initial soil was analysed for the mechanical composition and the soil belongs to clay loam texture with bulk density of  $1.31 \text{ Mg m}^{-3}$  and particle density of

$2.59 \text{ Mg m}^{-3}$  and porosity of 49.42%. The soil was sodic with a pH of 9.38 and EC of  $2.12 \text{ dSm}^{-1}$ . The exchangeable Na, Ca, Mg and CEC were 9.52, 4.93, 2.51 and  $21.6 \text{ C mol (p+) kg}^{-1}$  respectively. The Exchangeable Sodium P was calculated to 44.07 with a gypsum requirement of 12.66 tonnes  $\text{ha}^{-1}$ . The organic carbon content was  $4.60 \text{ g kg}^{-1}$  and the soil was low in available nitrogen ( $258 \text{ kg ha}^{-1}$ ), medium in Olsen's P ( $20 \text{ kg ha}^{-1}$ ) high in available potassium ( $354 \text{ kg ha}^{-1}$ ).

<b>I</b>	<b>Mechanical analysis</b>	
	Clay (%)	33.4
	Silt (%)	20.2
	Fine sand (%)	28.6
	Coarse sand (%)	17.0
	Textural class	Clay loam
	Soil series	Panjappur
<b>II</b>	<b>Physical constants</b>	
	Bulk density ( $\text{Mg m}^{-3}$ )	1.31
	Particle density ( $\text{Mg m}^{-3}$ )	2.59
	Pore space (%)	49.42
	Hydraulic conductivity ( $\text{cm hr}^{-1}$ )	0.44
<b>III</b>	<b>Physico-chemical properties</b>	
	pH (1:2.5 soil: water)	9.38
	EC ( $\text{dSm}^{-1}$ )	2.12
<b>IV</b>	<b>Chemical properties</b>	
	Organic carbon ( $\text{g kg}^{-1}$ )	4.60
	CEC ( $\text{C mol (p}^+) \text{ kg}^{-1}$ )	21.6
	Available nitrogen ( $\text{kg ha}^{-1}$ )	258
	Available phosphorus ( $\text{kg ha}^{-1}$ )	20.0
	Available potassium ( $\text{kg ha}^{-1}$ )	354
	Exchangeable sodium ( $\text{C mol (p}^+) \text{ kg}^{-1}$ )	9.52
	Exchangeable sodium percentage	44.07
	Exchangeable calcium ( $\text{C mol (p}^+) \text{ kg}^{-1}$ )	4.93
	Exchangeable magnesium ( $\text{C mol (p}^+) \text{ kg}^{-1}$ )	2.51
	Gypsum requirement ( $\text{tonnes ha}^{-1}$ )	12.66

### Characterization of nano-clay and nano-gypsum (Table 3)

**Physical Properties :Size:** The data revealed that the size of the nano-clay varied from 70 – 80 nm with an average dimension of 75 nm whereas that of synthesized nano gypsum varied from 80-98 nm with an average dimension of 89 nm .

**Shape:** The shape of the nano-clay was studied using high resolution Scanning Electron Microscopy (SEM) which shows the particles were irregular in shape. The synthesized nano-gypsum was also irregular in shape.

**Surface area:** The surface area of nano-clay was measured which exhibited higher surface area of  $1000 \text{ m}^2 \text{ g}^{-1}$  and that of synthesized nano-gypsum was  $900 \text{ m}^2 \text{ g}^{-1}$ .

**Bulk Density:** The bulk density of the nano-clay was  $0.45 \text{ g cm}^{-3}$  while increased by two times to  $0.48 \text{ g cm}^{-3}$  in synthesized nano-gypsum.

**Color:** The colour of the nano-clay was measured using Munsell colour chart. The results showed that nano-clay is dusty white in colour (5Y 8/1) whereas the colour of the synthesized nano-gypsum was light gray in colour (2.5 Y8/1).

<b>Parameters</b>	<b>Nano clay</b>	<b>Nano-gypsum</b>
<b>Physical Properties</b>		
Size (nm)	70-80 nm	80-98
Shape	Irregular	Irregular
Surface Area ( $\text{m}^2 \text{ g}^{-1}$ )	1000	900
Bulk Density ( $\text{g cm}^{-3}$ )	0.45	0.48
Color	5Y 8/1	2.5Y 8/1
<b>Chemical Properties</b>		
pH of 1% slurry	8.0	8.2
EC ( $\text{dSm}^{-1}$ )	0.02	0.34
CEC ( $\text{C mol (p}^+) \text{ kg}^{-1}$ )	187	189

**Chemical Properties:**

**pH:** The pH of the nano-clay was 8.0 and the pH of the synthesized nano-gypsum was 8.2.

**Electrical Conductivity (EC):** The electrical conductivity (EC) of the nano-clay was  $0.02 \text{ dSm}^{-1}$ , which was free from soluble salts, whereas the synthesized nano-gypsum has higher soluble salt content which measured  $0.14 \text{ dS m}^{-1}$ .

**Cation Exchange Capacity (CEC):** The Cation Exchange Capacity of the nano-clay was  $187 \text{ (C mol (p}^+) \text{ kg}^{-1})$  which was very high and that of synthesized nano-gypsum slightly increased to  $189 \text{ (C mol (p}^+) \text{ kg}^{-1})$ .

**Characterization of nano-clay(montmorillonite) before and after loading of gypsum:**

**SEM images:** The surface morphology of nano clay (montmorillonite) before and after loading with gypsum was examined under SEM. The result showed that nano-gypsum is very clustered consolidated with nano-clay (montmorillonite) is scattered and sparse.

**X-Ray Diffraction:** The XRD pattern of nano-clay (montmorillonite) before and after loading gypsum is given in the peaks at  $2\theta$  was observed for pure nano-clay (montmorillonite). When it was loaded with gypsum, there was change in peaks  $2\theta$ . There was a change in the peak angle and these peaks clearly showed that gypsum was attached on nano-clay.

**Raman spectrum:** The Raman spectrum studied for the nano-clay (montmorillonite) unloaded and loaded with gypsum. The relative intensities of the peaks were used to quantify the information on the composition of a mixture. The intensity of peaks for unloaded nano-clay (montmorillonite) were  $266.6, 1643.1, 1690.8, 1742.8$  and  $1781 \text{ cm}^{-1}$ . Gypsum carried the prominent peaks viz.,  $1006.5, 1034.5$  and  $1156 \text{ cm}^{-1}$ . The same way the peaks for nano-clay (montmorillonite) loaded with gypsum were  $271.7, 1742, 8, 1006.3, 1644.9, 1690$  and  $1781 \text{ cm}^{-1}$  which.

confirmed gypsum attachment with nano-clay **FT-IR**

**Spectra:** The IR spectra of unloaded nano-clay (montmorillonite) have characteristic peaks of  $3635.16, 2919.7, 1836.86, 1641.13, 1471.42, 1126.22, 799.35, 722.21, 625.788, 435.834$  and  $407.871 \text{ cm}^{-1}$ . The IR spectra of gypsum indicated that it had characteristic peaks of  $3306.36, 2237.02, 1686.44, 1626.66, 1237.11, 67.106, 605.539$  and  $428.12 \text{ cm}^{-1}$ . The IR spectra of nano-clay (montmorillonite) loaded with gypsum have characteristic peaks of  $3405.67, 2920.66, 2851.24, 2851.24, 2238.95, 1623.77, 1470.46, 1172.51, 799.35, 666.285, 600.717, 528.4$  and  $420.406 \text{ cm}^{-1}$ . There was a shift in the peaks after loading of gypsum. This confirmed that gypsum is attached into nano-clay (montmorillonite).

**Chemical analysis of the sodic soil after reclamation with nano-gypsum (Table 4)**

**Soil pH:** The pH of the soil was analysed after reclamation with nano-gypsum (NG) and conventional gypsum (CG). There was a profound change in the soil pH with addition of nano-gypsum and conventional gypsum at varying levels of application based on gypsum requirement (GR). The pH values ranged from 9.38 in control to 7.43 in the 100% GR as nano-gypsum applied treatments. Among the two sources, nano-gypsum recorded a pH of 8.41 and was significant over that recorded in conventional gypsum (8.78) irrespective of the levels. Among the different levels tried, application of 100% GR reduced the pH to 7.80 followed by 75% (8.20) and then 50% (8.60) and the effect of different levels was significant. However the interaction between the levels and sources was non-significant. A significant reduction in pH by the application of 100% GR as nano-gypsum (7.43) was recorded over the other levels and conventional gypsum.

**Table 4. Effect of nano gypsum /gypsum on soil pH and EC ( $\text{dSm}^{-1}$ )**

Treatments	pH			EC ( $\text{dSm}^{-1}$ )		
	NG	CG	Mean	NG	CG	Mean
Control	9.38	9.38	<b>9.38</b>	1.94	1.94	<b>1.94</b>
25% GR	8.89	9.09	<b>8.99</b>	1.72	1.74	<b>1.73</b>
50% GR	8.41	8.78	<b>8.60</b>	1.50	1.53	<b>1.52</b>
75% GR	7.92	8.47	<b>8.20</b>	1.28	1.39	<b>1.34</b>
100% GR	7.43	8.16	<b>7.80</b>	1.06	1.18	<b>1.12</b>
Mean	<b>8.41</b>	<b>8.78</b>	<b>8.59</b>	<b>1.50</b>	<b>1.56</b>	<b>1.53</b>
SEd	A	L	A×L	A	L	A×L
CD (0.05)	0.06	0.10	0.13	0.02	0.03	0.04
	0.12	0.205	NS	0.04	0.06	NS

**Soil EC:** The EC of the soil was significantly reduced by the application of the amendments. The least value of EC was recorded in 100% GR as nano-gypsum ( $1.06 \text{ dSm}^{-1}$ ) followed by 100% GR as conventional gypsum ( $1.18 \text{ dSm}^{-1}$ ). The application of 75% GR as nano-gypsum ( $1.28 \text{ dSm}^{-1}$ ) was on par with 100% conventional gypsum ( $1.18 \text{ dSm}^{-1}$ ) and 75% GR as conventional gypsum ( $1.39 \text{ dSm}^{-1}$ ) in reducing the EC. The significant effect of amendments and levels were observed

**Exchangeable sodium and Exchangeable Sodium Percentage (ESP) (Table 5):** Exchangeable sodium and exchangeable sodium percentage was significantly affected by the amendments. The exchangeable sodium was  $9.32 \text{ C mol (p}^+) \text{ kg}^{-1}$  in control and ESP was 43.15. The exchangeable sodium levels declined to  $2.24 \text{ C mol (p}^+) \text{ kg}^{-1}$  in 100% GR as nano-gypsum followed by  $3.18 \text{ C mol (p}^+) \text{ kg}^{-1}$  in 100% GR as conventional gypsum and  $3.14 \text{ C mol (p}^+) \text{ kg}^{-1}$  in 75% GR as nano-gypsum. The decline was significant over the control and other treatments. The application of 75% and 50% GR as conventional (14.72%).

gypsum recorded exchangeable sodium of 4.63 and  $6.20 \text{ C mol (p}^+) \text{ kg}^{-1}$  respectively. Among the two sources, nano-gypsum recorded the least exchangeable sodium of  $5.43 \text{ C mol (p}^+) \text{ kg}^{-1}$  which was significant over the application of conventional gypsum ( $6.22 \text{ C mol (p}^+) \text{ kg}^{-1}$ ). Among the levels 100% nano-gypsum significantly recorded the exchangeable sodium to  $2.71 \text{ C mol (p}^+) \text{ kg}^{-1}$  followed by 75% ( $3.89 \text{ C mol (p}^+) \text{ kg}^{-1}$ ).

The exchangeable sodium percentage being a measure of exchangeable sodium and CEC of the soil, a trend similar to that of exchangeable sodium was observed. The ESP significantly reduced to 12.55 in 100% GR as nano-gypsum treatment. The significant role of nano-gypsum in reducing the ESP to 25.15 was also recorded, while ESP reduced to 28.80 in the conventional gypsum treatments. The interaction among the amendments and levels was significant in both exchangeable sodium and ESP. The least value of 10.37 of ESP was recorded in 100% nano-gypsum. Application of 75% GR as nano-gypsum (14.54%) was on par with 100% GR conventional gypsum

Treatments	Na (C mol (p <sup>+</sup> ) kg <sup>-1</sup> )			ESP (%)		
	NG	CG	Mean	NG	CG	Mean
Control	9.32	9.32	<b>9.32</b>	43.15	43.15	<b>43.15</b>
25% GR	7.26	7.77	<b>7.52</b>	33.61	35.97	<b>34.79</b>
50% GR	5.20	6.20	<b>5.70</b>	24.07	28.70	<b>26.39</b>
75% GR	3.14	4.63	<b>3.89</b>	14.54	21.44	<b>17.99</b>
100% GR	2.24	3.18	<b>2.71</b>	10.37	14.72	<b>12.55</b>
Mean	<b>5.43</b>	<b>6.22</b>	<b>5.83</b>	<b>25.15</b>	<b>28.80</b>	<b>26.97</b>
SEd	A	L	A×L	A	L	A×L
CD (0.05)	0.07	0.12	0.16	0.34	0.54	0.77
	0.15	0.24	0.34	0.72	1.14	1.61

**Exchangeable cations in the soil (Table 6):** The exchangeable  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$  and  $\text{K}^+$  was analysed in the exchange complex to evaluate the extent of exchange of ions that has occurred during the reclamation process.

**Exchangeable calcium ( $\text{Ca}^{2+}$ ):** The exchangeable  $\text{Ca}^{2+}$  was  $4.93 \text{ C mol (p}^+) \text{ kg}^{-1}$  in control, while the exchangeable  $\text{Ca}^{2+}$  in the reclaimed soil increased up to  $13.2 \text{ C mol (p}^+) \text{ kg}^{-1}$ . The highest was recorded in soil amendment with 100% GR as nano-gypsum ( $13.2 \text{ C mol (p}^+) \text{ kg}^{-1}$ ). This was significantly higher than 100% GR as conventional gypsum ( $11.1 \text{ C mol (p}^+) \text{ kg}^{-1}$

<sup>1</sup>). Application of nano-gypsum significantly increased the exchangeable  $\text{Ca}^{2+}$  ( $9.08 \text{ C mol (p}^+) \text{ kg}^{-1}$ ) in the soil as against conventional gypsum ( $8.06 \text{ C mol (p}^+) \text{ kg}^{-1}$ ). Lower levels of exchange of  $\text{Ca}^{2+}$  were observed with reduction in the levels of amendments. The significant effect of amendments on the exchangeable  $\text{Ca}^{2+}$  was observed in the reclaimed soil.

**Exchangeable Magnesium ( $\text{Mg}^{2+}$ ):** There was no marked variation in the exchangeable  $\text{Mg}^{2+}$  of the soil due to amendments. However a significant decrease in  $\text{Mg}^{2+}$  from the control soil ( $2.5 \text{ C mol (p}^+) \text{ kg}^{-1}$ ) was recorded. Among the amendments, conventional

gypsum recorded significantly higher exchangeable  $Mg^{2+}$  of 2.4 C mol ( $p^+$ )  $kg^{-1}$  over nano-gypsum and among the levels, 100% , 75% and 50% GR were on par.

**Exchangeable Potassium:** The effect of amendments and their levels was non significant with respect to exchangeable potassium of the reclaimed soil. The results on the exchangeable cations in the soil viz.,  $Ca^{2+}$ ,  $Mg^{2+}$  and  $K^+$  after reclamation revealed the significant effect of both conventional gypsum and nano-gypsum in reclaiming the sodic soil.

The higher concentration of  $Ca^{2+}$  ions in the exchange complex proved the positive effect of nano-gypsum and conventional gypsum in the reclamation of sodic soil. However the soil treated with nano-gypsum has accumulated more  $Ca^{2+}$  in the exchange complex than conventional gypsum. The significant effect of nano-gypsum in replacing  $Na^+$  from the complex over that of conventional gypsum could be attributed to the finer particle size, higher CEC and solubility of nano-

gypsum. The exchangeable  $Ca^{2+}$  increased with increasing levels of gypsum irrespective of sources confirming its ameliorating property. Similar findings were also reported by Patel and Suthar (1993) and Ilyaset *al.* (1997). The increase in the concentration of  $Ca^{2+}$  in the exchange sites with the increasing levels of amendments applied indicated that, higher the quantity of  $Ca^{2+}$  supplied as  $CaSO_4 \cdot 2H_2O$  , higher the replacing power and reclamation.

With respect to  $Mg^{2+}$  ions, though there was no distinct variation among the treatments, a significant increase over the control was registered, which might be due to replacement of  $Mg^{2+}$  from the exchange sites. In general, a drastic change could not be recorded as no magnesium source was added.

A smaller but non significant decrease in exchangeable  $K^+$  was recorded which might be due to the reason that  $Ca^{2+}$  would have occupied the exchange sites of  $K^+$  in the soil during the reclamation process.

Treatments	Ca ( C mol ( $p^+$ ) $kg^{-1}$ )			Mg ( C mol ( $p^+$ ) $kg^{-1}$ )			K ( C mol ( $p^+$ ) $kg^{-1}$ )		
	NG	CG	Mean	NG	CG	Mean	NG	CG	Mean
<b>Control</b>	4.93	4.93	<b>4.93</b>	2.48	2.48	<b>2.48</b>	4.51	4.51	<b>4.51</b>
<b>25% GR</b>	7.03	6.52	<b>6.78</b>	2.40	2.42	<b>2.41</b>	4.45	4.47	<b>4.46</b>
<b>50% GR</b>	9.09	8.09	<b>8.59</b>	2.31	2.37	<b>2.34</b>	4.41	4.44	<b>4.43</b>
<b>75% GR</b>	11.2	9.66	<b>10.4</b>	2.28	2.32	<b>2.30</b>	4.38	4.42	<b>4.40</b>
<b>100% GR</b>	13.2	11.1	<b>12.2</b>	2.21	2.29	<b>2.25</b>	4.33	4.40	<b>4.37</b>
<b>Mean</b>	<b>9.08</b>	<b>8.06</b>	<b>8.57</b>	<b>2.34</b>	<b>2.38</b>	<b>2.36</b>	<b>4.42</b>	<b>4.45</b>	<b>4.43</b>
<b>S<sub>Ed</sub></b>	<b>A</b>	<b>L</b>	<b>A×L</b>	<b>A</b>	<b>L</b>	<b>A×L</b>	<b>A</b>	<b>L</b>	<b>A×L</b>
<b>CD (0.5)</b>	0.09	0.14	0.19	0.02	0.04	0.05	0.05	0.07	0.10
	0.19	0.29	0.41	NS	0.08	NS	NS	NS	NS

**Concentration of ions in the leachate (Tables 7-8):** The leachate that was collected during the reclamation process was analysed for the concentration of ions to have knowledge on the nature and quantity of ions that were leached.

**Sodium concentration :** The leachate contained 191 ppm of  $Na^+$  in the soil reclaimed with 100% GR as nano-gypsum followed by 144 ppm in 75% GR as nano-gypsum and 143 ppm in 100% GR as conventional gypsum and 110 ppm in 75% GR as conventional gypsum. The least concentration of 1.84 ppm was measured in the control.

Among the sources, nano-gypsum leached out a maximum of 96.6 ppm of Na which was significant over conventional gypsum (73.1 ppm). Among the levels, application of 100% GR of amendments irrespective of the sources leached out 167 ppm of Na followed by 127 ppm in 75% GR.

**Calcium concentration :** The concentration of  $Ca^{2+}$  ions in the leachate ranged from 0.80 ppm in the control to 27.6 ppm in 100% GR as conventional gypsum. Among the two sources of amendments, conventional gypsum leached out more of  $Ca^{2+}$  (14.0 ppm) than nano-gypsum (9.84 ppm). At all levels tried, the amount of  $Ca^{2+}$  ions was lesser in the soil

reclaimed with nano-gypsum than with conventional gypsum. Irrespective of the sources, the leachate from 100% GR treated sodic soil registered higher Ca<sup>2+</sup> (23.7 ppm) followed by 75% GR (17.4 ppm), 50% GR (12 ppm), 25% GR (5.60 ppm) and control (0.80 ppm).

**Magnesium concentration:** The concentration of magnesium (Mg<sup>2+</sup>) in the leachate was very low ranging from 0.36 ppm to 3.60ppm. A trend similar to that of Ca<sup>2+</sup> was observed in the concentration of Mg<sup>2+</sup> in the leachate.

**Potassium concentration :** The nano-gypsum applied sodic soil leached out more of K<sup>+</sup> (4.45 ppm) and this was significantly higher than conventional gypsum (3.20 ppm). Similarly, the higher levels of amendments leached out higher concentration of K<sup>+</sup> (6.44 ppm) and this was superior to the other levels. The mean values of K<sup>+</sup> concentration in the leachate ranged from 0.78 ppm in control to 7.80 ppm in the 100% GR as nano-gypsum.

**Table 7. Concentration of cations in the leachate**

Treatments	Na (me L <sup>-1</sup> )			Ca <sup>2+</sup> (meL <sup>-1</sup> )			Mg <sup>2+</sup> (me L <sup>-1</sup> )			K <sup>+</sup> (me L <sup>-1</sup> )		
	NG	CG	Mean	NG	CG	Mean	NG	CG	Mean	NG	CG	Mean
<b>Control</b>	0.08	0.08	<b>0.08</b>	0.04	0.04	<b>0.04</b>	0.03	0.03	<b>0.03</b>	0.02	0.02	<b>0.02</b>
<b>25% GR</b>	2.14	1.63	<b>1.89</b>	0.21	0.35	<b>0.28</b>	0.11	0.09	<b>0.10</b>	0.08	0.06	<b>0.07</b>
<b>50% GR</b>	4.20	3.20	<b>3.70</b>	0.46	0.74	<b>0.60</b>	0.20	0.14	<b>0.17</b>	0.12	0.09	<b>0.11</b>
<b>75% GR</b>	6.26	4.77	<b>5.52</b>	0.76	0.98	<b>0.87</b>	0.23	0.19	<b>0.21</b>	0.15	0.11	<b>0.13</b>
<b>100% GR</b>	8.32	6.22	<b>7.27</b>	0.99	1.38	<b>1.19</b>	0.30	0.22	<b>0.26</b>	0.20	0.13	<b>0.17</b>
<b>Mean</b>	<b>4.20</b>	<b>3.18</b>	<b>3.69</b>	<b>0.49</b>	<b>0.70</b>	<b>0.60</b>	<b>0.17</b>	<b>0.13</b>	<b>0.15</b>	<b>0.11</b>	<b>0.08</b>	<b>0.10</b>
<b>SEd</b>	A	L	A×L	A	L	A×L	A	L	A×L	A	L	A×L
<b>CD (0.05)</b>	0.05	0.10	0.12	0.01	0.01	0.02	0.001	0.002	0.004	0.001	0.001	0.001
	0.11	0.20	0.25	0.01	0.03	0.03	0.003	0.006	0.008	0.002	0.002	0.003

**Carbonate and bicarbonate concentration in the leachate:** Higher concentration of HCO<sub>3</sub><sup>-</sup> was leached out than the CO<sub>3</sub><sup>2-</sup> ions in the treatments. However, the leaching of both the ions followed a similar trend. The application of nano gypsum leached out more of CO<sub>3</sub><sup>2-</sup> ions (6.30 ppm) which was significant over conventional gypsum (4.78 ppm).The HCO<sub>3</sub><sup>-</sup> concentration was 141 ppm in nano-gypsum treatments and this was significant over that of conventional gypsum. The leachate from the untreated (control) sodic soil recorded 0.12 ppm of CO<sub>3</sub><sup>2-</sup> and 2.68 ppm of HCO<sub>3</sub><sup>-</sup> ions.

**Chlorides concentration (Cl):** The leachate contained chlorides ranging from 0.86 ppm to 88.7

ppm. Significantly higher concentration was recorded in 100 %GR as nano gypsum followed by 100% GR as conventional gypsum and 75% GR as nano gypsum. The application of 75% nano gypsum was on par with 100% GR as conventional gypsum.

**Sulphate concentration (SO<sub>4</sub><sup>2-</sup>):** The concentration of SO<sub>4</sub><sup>-</sup> ions in the leachate ranged from 4.70 ppm to 116 ppm. The concentration in the leachate due to the two sources of amendments was non significant. Among the levels, 100% GR leachate out higher levels of SO<sub>4</sub><sup>2-</sup> (114 ppm) and reduced with decreasing levels of GR applied.

**Table 8. Concentration of anions in the leachate**

Treatments	Co <sub>3</sub> <sup>2-</sup> (me L <sup>-1</sup> )			HCO <sub>3</sub> <sup>-</sup> (me L <sup>-1</sup> )			Cl <sup>-</sup> (me l <sup>-1</sup> )			SO <sub>4</sub> <sup>2-</sup> (me L <sup>-1</sup> )		
	NG	CG	Mean	NG	CG	Mean	NG	CG	Mean	NG	CG	Mean
<b>Control</b>	0.00	0.00	<b>0.00</b>	0.04	0.04	<b>0.04</b>	0.02	0.02	<b>0.02</b>	0.10	0.10	<b>0.10</b>
<b>25% GR</b>	0.11	0.08	<b>0.09</b>	1.18	0.90	<b>1.04</b>	0.64	0.49	<b>0.57</b>	0.61	0.66	<b>0.64</b>
<b>50% GR</b>	0.21	0.16	<b>0.19</b>	2.31	1.76	<b>2.04</b>	1.26	0.96	<b>1.11</b>	1.20	1.29	<b>1.25</b>
<b>75% GR</b>	0.31	0.24	<b>0.28</b>	3.44	2.62	<b>3.03</b>	1.88	1.43	<b>1.66</b>	1.77	1.78	<b>1.78</b>
<b>100% GR</b>	0.42	0.31	<b>0.36</b>	4.58	3.42	<b>4.00</b>	2.50	1.87	<b>2.19</b>	2.42	2.35	<b>2.39</b>
<b>Mean</b>	<b>0.21</b>	<b>0.16</b>	<b>0.18</b>	<b>2.31</b>	<b>1.75</b>	<b>2.03</b>	<b>1.26</b>	<b>0.95</b>	<b>1.11</b>	<b>1.22</b>	<b>1.24</b>	<b>1.23</b>
<b>SEd</b>	A	L	A×L	A	L	A×L	A	L	A×L	A	L	A×L
<b>CD (0.05)</b>	0.003	0.004	0.01	0.02	0.03	0.05	0.01	0.02	0.03	0.01	0.02	0.03
	0.01	0.01	0.011	0.05	0.08	0.11	0.03	0.04	0.06	NS	0.04	0.05

The interaction among the sources and levels were significant with respect to the sulphate concentration

in the leachate. The concentration of Na<sup>+</sup> in the leachate of the control

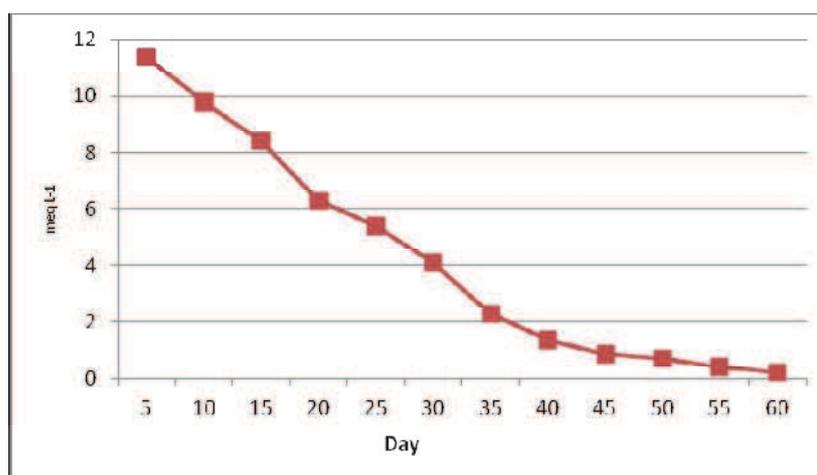
sodic soil vividly depicts that soil exchange complex was loaded with  $\text{Na}^+$ . In the leachate collected from the different treatments, the  $\text{Na}^+$  replacement was well observed in the soil reclaimed with nano-gypsum and conventional gypsum. The higher concentration of  $\text{Na}^+$  leached out by the addition of nano-gypsum indicates the reclamative property of nano-gypsum. At all levels of conventional gypsum added to remediate the sodic soil, the quantity of  $\text{Ca}^{2+}$  leached was more than the corresponding nano-gypsum, thus reflecting the retaining capacity of the smaller sized nano-gypsum for  $\text{Ca}^{2+}$ . Hanayet *et al.* (2004) also reported the significant effect when gypsum was reduced to 0.149 mm in size.

There was very little leaching of  $\text{Mg}^{2+}$  recorded in all the treatments, but the effect of the amendments was similar to that of  $\text{Ca}^{2+}$ . The quick action of gypsum (either as conventional gypsum or nano-gypsum) in dissolving the insoluble salts and flushing out the  $\text{Na}^+$  ions was registered. The increase in  $\text{Na}^+$  concentration in the leachate depicts clearly the steady reaction of the amendments with sodium to replace it. Similar results were reported by Sabiret *et al.* (2007).

Similar effect had been observed for the associated anions. However, the concentration of anions viz.,

$\text{HCO}_3^-$  and  $\text{CO}_3^{2-}$  were higher than the chlorides and sulphates. Singh *et al.* (2011) reported that the  $\text{Ca}^{2+}$  of gypsum displaced  $\text{Na}^+$  from the exchange sites and removal of sodium with the anions viz.,  $\text{HCO}_3^-$  and  $\text{CO}_3^{2-}$  through leaching reduced the pH of the soil. The similar trend in the concentration of anions as that of  $\text{Na}^+$  in the leachate indicates the removal of associated anions viz.,  $\text{HCO}_3^-$ ,  $\text{CO}_3^{2-}$ ,  $\text{Cl}^-$ , and  $\text{SO}_4^{2-}$  by ponding and surface drainage of the reclaimed sodic soil. Earlier work by Mishra *et al.* (2003) also reported similar results in the leachate collected during the reclamation of sodic soil. Abbas *et al.* (2004) reported that  $\text{Cl}^-$  and  $\text{HCO}_3^-$  were easily leached from the soil.

**Percolation study to assess the efficiency of nano-gypsum:** The efficiency of nano-gypsum was examined using the percolation reactor. The data on  $\text{Ca}^{2+}$  of the leachate clearly showed that, the  $\text{Ca}^{2+}$  content declined proportionately with the progress of the experiment. Initially, the  $\text{Ca}^{2+}$  content measured was in the range of 8-10  $\text{meq L}^{-1}$ , while in the last stage of the experiment (55-60 days), the  $\text{Ca}^{2+}$  content has reduced to the minimum of 0.2 to 0.4  $\text{meq L}^{-1}$ . The  $\text{Ca}^{2+}$  concentration almost ceased to exist in the leachate at the end of the experimental period (Fig.1).



**Fig.1 Calcium as released from nano-gypsum (Percolation study)**

Nano-gypsum possesses extensive surface area and adsorptive sites to exchange  $\text{Ca}^{2+}$  with  $\text{Na}^+$  in the soil exchangeable complex. Since nano-gypsum efficiently retained  $\text{Ca}^{2+}$  and displaced  $\text{Na}^+$ , the  $\text{Ca}^{2+}$  content of the leachate linearly declined with the progress of the experiment. The study further indicates a long lasting effect of nano-gypsum to regulate the retention of  $\text{Ca}^{2+}$  in the clay complex, while having considerable low amounts of  $\text{Ca}^{2+}$  in the solution. The data corroborates with the observations of our pot culture experiment, where the leachate of surface drainage were determined for the concentration  $\text{Ca}^{2+}$  and  $\text{Na}^+$  ions.

The application of nano-gypsum @75% GR also proved effective as it was comparable to 100% GR as conventional gypsum in all the parameters confirming reclamation. Among the amendments, nano-gypsum proved its efficacy in reclaiming the sodic soils significantly than conventional gypsum which could be attributed to its smaller particle size, larger surface area and there by greater solubility. Application of 100% GR as nano-gypsum conspicuously reclaimed the sodic soil. However, 75% GR as nano-gypsum was comparable to 100% GR as conventional gypsum. The prominent effect of increasing doses of amendments was also observed in the study.

It is concluded from the present study that, nano-

gypsum can be thought of as a viable alternative to conventional gypsum to reclaim sodic soil either at 100% or 75 % GR.

Further research is needed to ascertain the effectiveness of nano-gypsum at field levels, sustenance of reclamation affected by nano-gypsum, ,

the effective mixing ratio of nano-clay and gypsum, the reaction of nano- gypsum in the soil, retaining capacity of  $\text{Ca}^{2+}$  by nano gypsum, the effective depth of application nano-gypsum to the soil at field level and the scope for large scale production of nano-gypsum.

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