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**Gunavathi P**

Department of Soil Science and Agricultural Chemistry, Anbil Dharmalingam Agricultural College and Research Institute, Trichy, Tamil Nadu, India

**Janaki D**

Department of Soil Science and Agricultural Chemistry, Anbil Dharmalingam Agricultural College and Research Institute, Trichy, Tamil Nadu, India

**Balasubramaniam P**

Department of Soil Science and Agricultural Chemistry, Anbil Dharmalingam Agricultural College and Research Institute, Trichy, Tamil Nadu, India

**Alagesan A**

Department of Soil Science and Agricultural Chemistry, Anbil Dharmalingam Agricultural College and Research Institute, Trichy, Tamil Nadu, India

**Geethanjali S**

Department of Soil Science and Agricultural Chemistry, Anbil Dharmalingam Agricultural College and Research Institute, Trichy, Tamil Nadu, India

**Corresponding Author:**

**Gunavathi P**

Department of Soil Science and Agricultural Chemistry, Anbil Dharmalingam Agricultural College and Research Institute, Trichy, Tamil Nadu, India

## Characterization and identification of elemental sulphur, iron pyrite, mineral gypsum, Phospho gypsum and marine gypsum by using ATR-FTIR

**Gunavathi P, Janaki D, Balasubramaniam P, Alagesan A and Geethanjali S**

### Abstract

The Amendments generally used for sodic soil reclamation should be source of sulphates viz., Elemental sulphur, Iron pyrite, Mineral gypsum, Phospho gypsum and Marine gypsum. Characterization of sources by ATR-FTIR is rapid and elementary. The peaks of IR spectra obtained from FTIR is compared with Bio-Rad (Know it all) software, to interpret and qualitative analysis of functional groups in mentioned materials. The active peaks of spectra denote the presence of principle compounds of sulphur as well as gypsum. ATR-FTIR is most promising and non destructive method to precisely provide the result without tiresome conventional chemical analysis of characterization. The strong spectra of gypsum held at  $668\text{cm}^{-1}$ ,  $1130\text{cm}^{-1}$ ,  $1630\text{cm}^{-1}$  and  $2200\text{cm}^{-1}$  range. The OH stretch at  $3522\text{cm}^{-1}$  and  $3400\text{cm}^{-1}$  confirmed the presence of gypsum and sulphate compounds. The S-O bending of gypsum observed at  $669\text{cm}^{-1}$  and  $597\text{cm}^{-1}$ . The S-O stretching and S-O bending of sulphates peaks appeared at  $1140-1080\text{cm}^{-1}$  and  $680-610\text{cm}^{-1}$ . The alkyl thioketones ( $\text{-C=S}$ ) of sulphur compounds vibrations found at the range of  $1240-1050\text{cm}^{-1}$ . This helps to confirm that the sources are suitable for sodic soil reclamation.

**Keywords:** ATR-FTIR, Sulphur, Mineral gypsum, Phospho gypsum, Marine gypsum

### Introduction

The available materials which are used for sodic soil reclamation is source of calcium and sulphur compounds. The characterization and analysis of such materials by chemical methods are tedious and destructive. The FTIR instrumental analysis is rapid, error free and non destructive. The chemicals using for conventional analysis will invade and sometimes produce invalid results. Even with micro quantities of sample, it provides highly precise and reliable data. The IR spectroscopy draws attention due to identification and analysis of multi-components as well as minor and traces in systematic manner (Wei, *et al.*, 2016) (La Russa, *et al.*, 2009) [17, 9]. Sulfate mode frequencies such as  $\nu_1$ ,  $\nu_2$ ,  $\nu_3$ , and  $\nu_4$  are similar for all Ca-Sulfates spectra. And a broad peak is observed in  $\nu_2$  vibration for gypsum (Bishop *et al.*, 2014) [3]. Identification and pinpointing of functional groups in sodic soil reclamation sources will provide information about chemistry and nature of those materials. The objective of the study is to present and discuss spectra of sodic soil reclamation sources such as Elemental sulphur, Iron pyrite, Mineral gypsum, Phospho gypsum and Marine gypsum.

### Materials and Methods

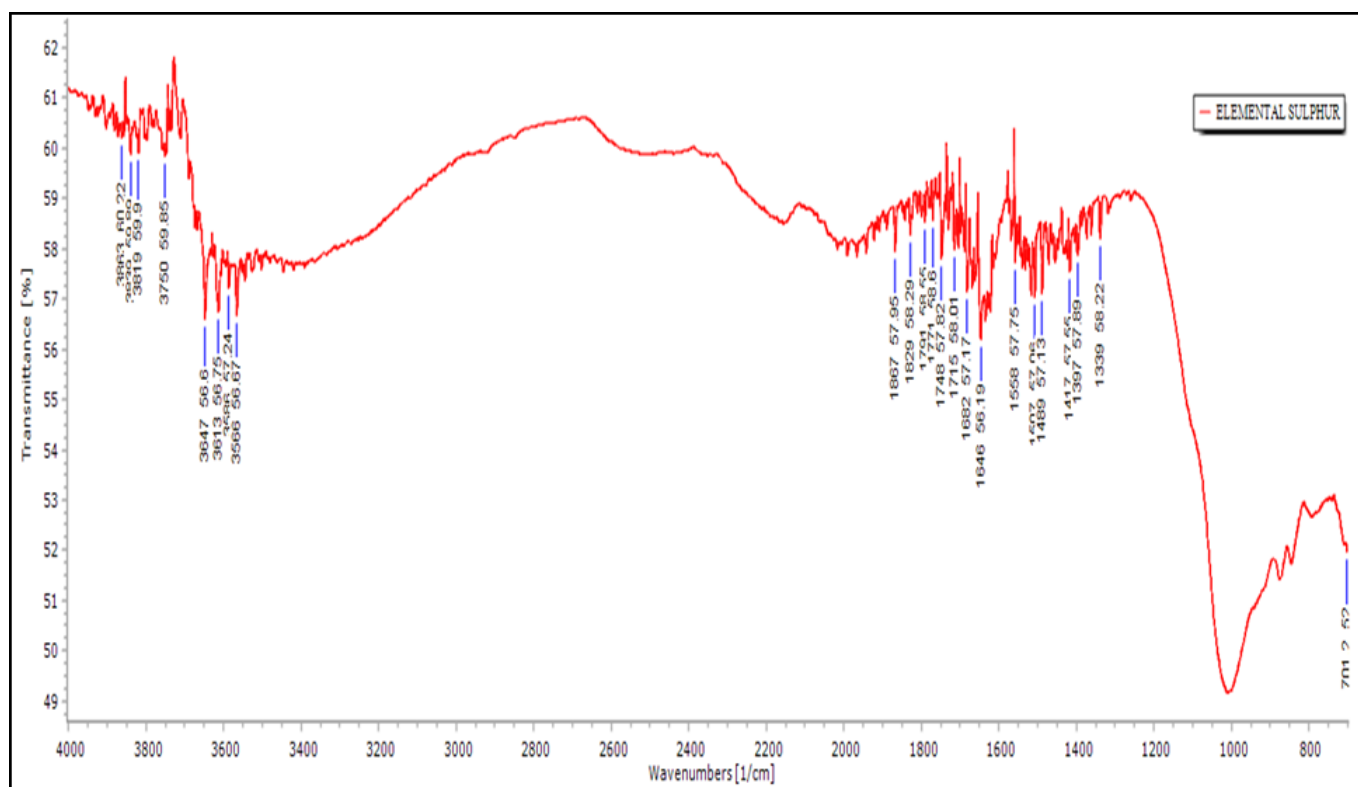
Fourier Transformer Infra Red Spectroscopy (FTIR) of Thermo Scientific nicole IS10 accompanied with omnic software is used to identify the spectrum of materials used for sodic soil reclamation such as Elemental sulphur, Iron pyrite, Mineral Gypsum, Phospho gypsum and Marine gypsum. Pinch of fine powdered samples were placed on diamond platform of attenuated total reflectance (ATR-FTIR). ATR-FTIR is ease and promising non destructive method of characterizing samples. It is an alternative way for transmission spectroscopy. The Infra red beam of high optical density passed through samples by critical angle, absorbed waves are reflected and recorded by detector (Blum MM and John H, 2011). Within 30-40 seconds of contact, the spectral data is generated. The spectra obtained from omnic software is processed by Bio-Rad (Know it all) software, which comprises various spectral library (Asan Mohamed and Janaki, 2021) [2].

## Results and Discussion

### Elemental Sulphur

The functional groups identified from Elemental Sulphur are Triazines, Azo and Silicon compounds. Coupling or overlapping of Triazines and Azo compounds found at the region of 1550-1400 $\text{cm}^{-1}$ . Hence it forms weak vibration stretching. Under the silicon compounds, strong and sharp Si-H stretching is obtained at 2160-2120 $\text{cm}^{-1}$ . Likewise, two sharp and strong bands of Ar-Si aromatic silanes obtained at 1430-1410  $\text{cm}^{-1}$  and 1120-1100  $\text{cm}^{-1}$ . Medium intensity of Si-CH<sub>3</sub> asymmetric deformation vibration held at range of 1420-1390  $\text{cm}^{-1}$ . Symmetrically, strong and sharp deformation vibration of Si-CH<sub>3</sub> group found near 1260-1230  $\text{cm}^{-1}$ . Strong

and broad stretch of Si-C is located at 900-700  $\text{cm}^{-1}$  in Spectral peak (Fig.1). Sulfate minerals may be distinguished using thermal infrared (mid-infrared) spectroscopy. The infra red spectra of aqueous sulfate anion shows symmetrical stretch ( $\nu_1$ ) at peak 983 $\text{cm}^{-1}$ . These vibrations are modified when the sulfate anion is present within a solid-state medium, such as a mineral with a repeating molecular order, resulting in the potential appearance of sulfate vibration modes in the spectrum (Lane M, 2007) [10]. The weak to medium absorbance symmetrical stretching of sulfone (SO<sub>2</sub>-C) peak found at 1100-1126 $\text{cm}^{-1}$ . The peak of Thiophene shows weak ring stretching at 1395-1412 $\text{cm}^{-1}$  (Tang *et al.*, 2020) [16].



**Fig 1:** IR Spectra of Elemental sulphur

### Iron Pyrite

The spectral band of Iron Pyrite shows the functional groups such as Alkanes, Triazines and Azo compounds. The C-H stretching vibration of normal alkanes has CH<sub>3</sub> asymmetric and symmetric stretching at 2972-2952 $\text{cm}^{-1}$  and 2882-2862 $\text{cm}^{-1}$  respectively. Asymmetrical and symmetrical stretch of CH<sub>2</sub> at ranges of 2936-2916 $\text{cm}^{-1}$  and 2863-2843 $\text{cm}^{-1}$ . The overlapped asymmetrical bending of CH<sub>3</sub> and CH<sub>2</sub> IN C-H bending vibrations located at 1470-1430 $\text{cm}^{-1}$  and 1485-1445  $\text{cm}^{-1}$ . The carbon attached CH<sub>3</sub> symmetrical bending of C-H bending vibrations located at 1380-1365 $\text{cm}^{-1}$ . C-H weak wagging vibration held at 1307-1303 $\text{cm}^{-1}$  and rocking

vibration of CH<sub>2</sub> at 750-722 $\text{cm}^{-1}$ . Alkanes of Mono, Di, Tri, Tetra and Penta substituted benzene compounds held at range of 3100-675 $\text{cm}^{-1}$ . The triazines of weak N=N stretching vibrations (aromatic azo) is obtained at 1550-1400 $\text{cm}^{-1}$ . The azo compounds also arising from the aromatic ring vibrations give weak vibration due to coupling or overlapping (Fig.2). The symmetric stretching of sulfate appears at near 1011 $\text{cm}^{-1}$  and 996  $\text{cm}^{-1}$ . The maximum bands of SO<sub>4</sub> mode observed in the region of 1000-1300 $\text{cm}^{-1}$  (Rouchon *et al.*, 2012) [15]. The peaks of pyrite (Fe=S), stretching of S-S and Fe-S arising in the range around 1120-1156 $\text{cm}^{-1}$  and 607-622 $\text{cm}^{-1}$  (Khabbaz and Entezari, 2016) [8].

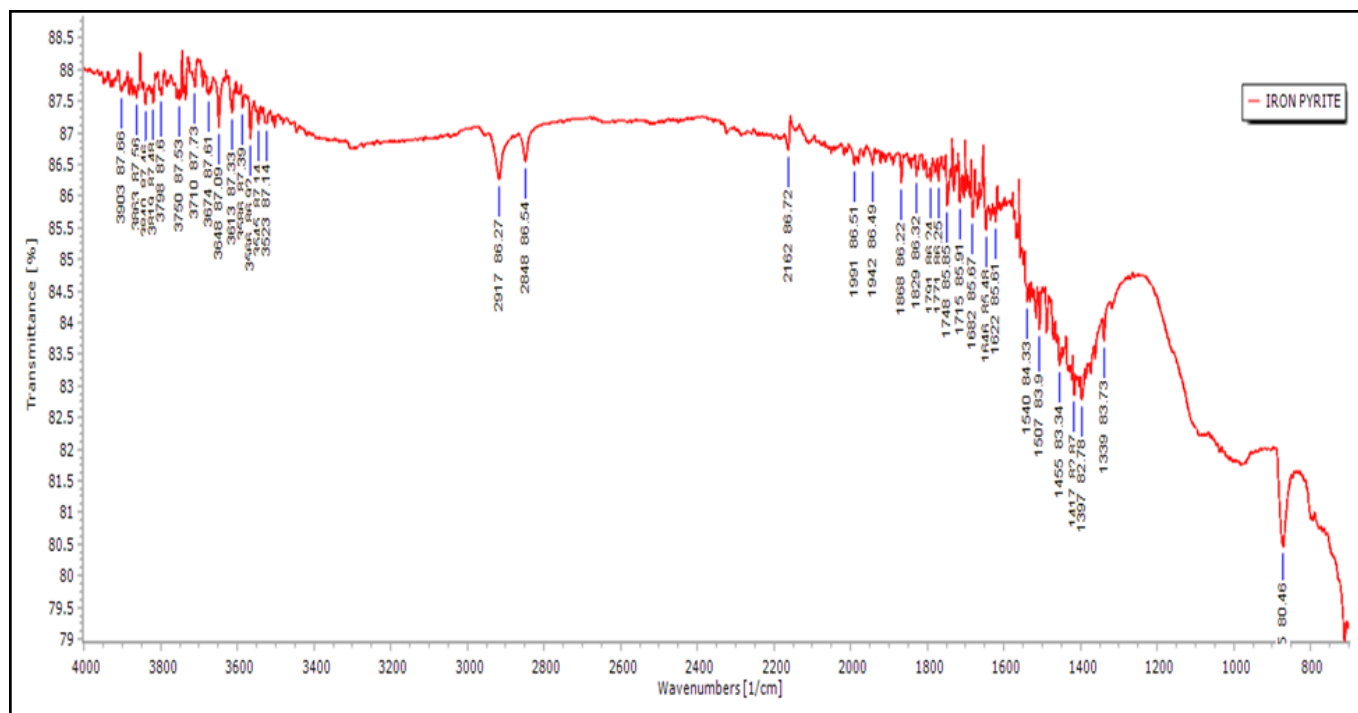


Fig 2: IR Spectra of Iron pyrite

### Mineral gypsum

The alcohols, nitriles, alkynes, ethers, halogens, phosphorous, silicon and sulphur compounds are identified in mineral gypsum spectral peaks. The primary, secondary and tertiary alcohols of strong intense O-H stretching, C-OH stretching and O-H deformation vibrations held at range of 3650-3450 $\text{cm}^{-1}$ , 1215-970 $\text{cm}^{-1}$  and near 1400 $\text{cm}^{-1}$  respectively. The sharp, weak to strong nitriles (C=N) stretching vibration found at 2260-2240 $\text{cm}^{-1}$  which is conjugated with olefinic C=C and aromatic C=C resulting lowered frequency in absorption band and intensity of C=N absorption band. The mono substituted alkynes (RC=CH) vibrations held at range of 3300-615 $\text{cm}^{-1}$  and di substituted alkynes (RC=CR') of non-conjugated stretching found at 2260-2190 $\text{cm}^{-1}$ . The strong multiple absorption bands of aliphatic ethers and aromatic ethers with side chained oxygen to C atom found at 1225-1060 $\text{cm}^{-1}$  and the aromatic C-O-C of ethers stretching in vibration gives strong and medium absorption band at 1270-1230 $\text{cm}^{-1}$  and 1050-1010 $\text{cm}^{-1}$  and its heterocyclic bands obtained at 1250-830 $\text{cm}^{-1}$ . In halogens, fluorinated aliphatic hydrocarbons and fluorinated aromatic hydrocarbons are found in the range of 1400-1000 $\text{cm}^{-1}$  and 1300-1200 $\text{cm}^{-1}$  respectively. The P-H, alkyl phosphines, aryl phosphines, cyclic and acyclic P=N compounds shows weak to medium intensity spectra at 2500-965 $\text{cm}^{-1}$ , 2285-910 $\text{cm}^{-1}$ , 2285-1085 $\text{cm}^{-1}$  and 1500-1170 $\text{cm}^{-1}$

respectively. The aliphatic ethers and aromatic ethers of silicon compounds gives strong multiple absorption band at 1225-1060 $\text{cm}^{-1}$  and the aromatic C-O-C of ethers stretching in vibration gives strong and medium absorption band at 1270-1230 $\text{cm}^{-1}$  and 1050-1010 $\text{cm}^{-1}$  and its heterocyclic bands obtained at 1100-900 $\text{cm}^{-1}$ . The alkyl thioketones (-C=S) of sulphur compounds vibrations found at the range of 1240-1050 $\text{cm}^{-1}$  (Fig.3). The alcohol and hydroxyl compounds of dimeric, internally bonded, non bonded, primary alcohol, secondary alcohol, tertiary alcohol and phenols are observed in peaks of 3550-3450 $\text{cm}^{-1}$ , 3570-3540 $\text{cm}^{-1}$ , 3645-3600 $\text{cm}^{-1}$ , 3645-3630 $\text{cm}^{-1}$ , 3635-3620 $\text{cm}^{-1}$ , 3620-3540 $\text{cm}^{-1}$  and 3640-3530 $\text{cm}^{-1}$  respectively (Nandiyanto *et al.*, 2019) (Coates J, 2000) [12, 6]. The C-F stretching is observed at 1340  $\text{cm}^{-1}$  in ATR-FTIR (Petit T and Ljiljana Puskar, 2018) [14]. The strong spectra of gypsum held at 668 $\text{cm}^{-1}$ , 1130 $\text{cm}^{-1}$ , 1630 $\text{cm}^{-1}$  and 2200 $\text{cm}^{-1}$  range. And the weak bands are arising at 1010 $\text{cm}^{-1}$  and 1670 $\text{cm}^{-1}$ . Sulfate mode frequencies such as  $\nu_1$ ,  $\nu_2$ ,  $\nu_3$ , and  $\nu_4$  are similar to calcium sulfates data obtained from transmission, Raman and emission spectra (Bishop *et al.*, 2014) [3]. The spectrum of pure gypsum which shows centered strong bands of sulfate at 1140 $\text{cm}^{-1}$  and 1128 $\text{cm}^{-1}$  and the strong peaks of sulfate shown in ranges of 669 $\text{cm}^{-1}$  and 602  $\text{cm}^{-1}$  (AL Dabbas *et al.*, 2014) [1].

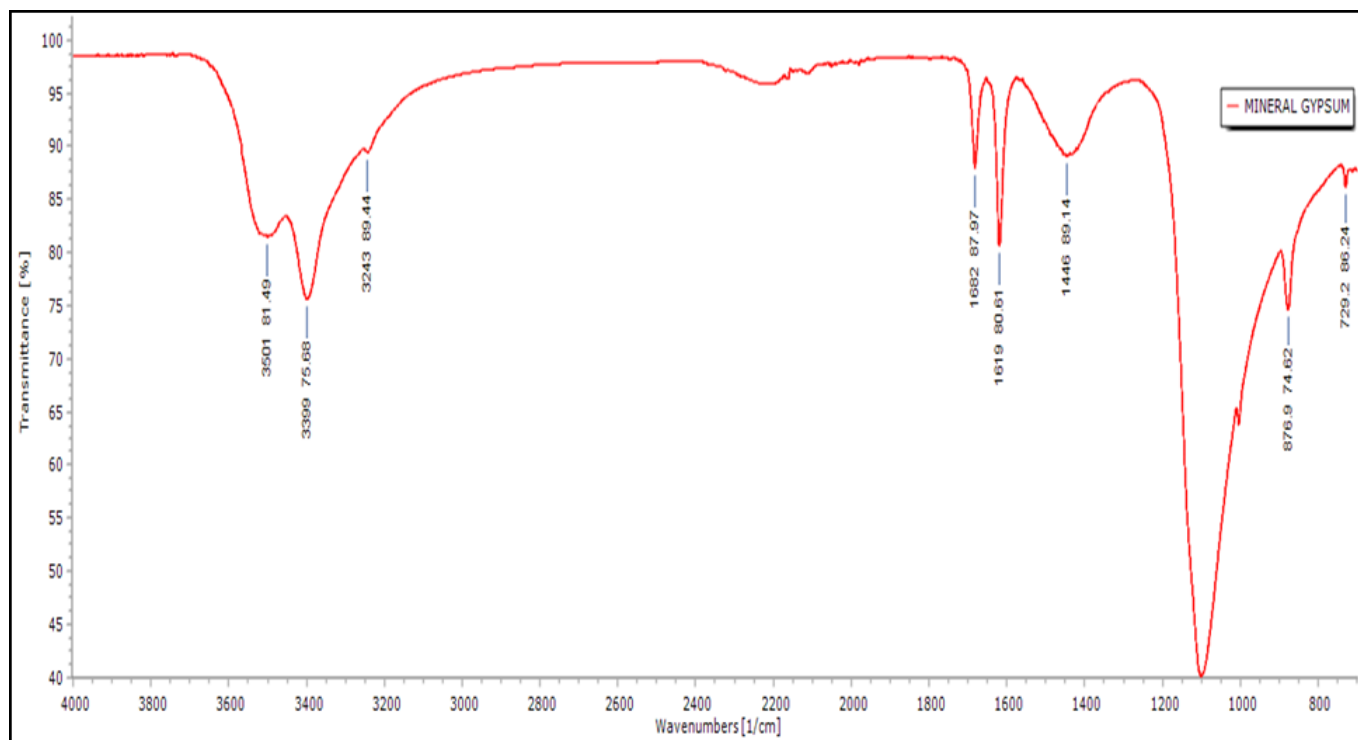


Fig 3: IR Spectra of Mineral Gypsum

#### Phospho gypsum:

The alcohols, nitriles, alkynes, ethers, halogens, phosphorous, silicon and sulphur compounds are identified in mineral gypsum spectral peaks. The strong intense O-H stretching of primary, secondary and tertiary alcohols arising at 3650-3320 $\text{cm}^{-1}$ , The C-OH stretching of primary, secondary and tertiary alcohols held at range of 1075-1000 $\text{cm}^{-1}$ , 1125-970 $\text{cm}^{-1}$  and 1210-1100 $\text{cm}^{-1}$ . The O-H deformation vibrations found at near 1400 $\text{cm}^{-1}$ . The sharp, weak to strong nitriles(C=N) stretching vibration found at 2260-2240 $\text{cm}^{-1}$  which is conjugated with olefinic C=C and aromatic C=C resulting lowered frequency in absorption band and intensity of C=N absorption band. The mono substituted alkynes (RC=CH) vibrations held at range of 3300-615 $\text{cm}^{-1}$  and di substituted alkynes (RC=CR') of non-conjugated stretching found at 2260-2190 $\text{cm}^{-1}$ . The strong multiple absorption bands of aliphatic ethers and aromatic ethers with side chained oxygen to C atom found at 1225-1060 $\text{cm}^{-1}$  and the aromatic C-O-C of ethers stretching in vibration gives strong and medium absorption band at 1270-1230 $\text{cm}^{-1}$  and 1050-1010 $\text{cm}^{-1}$  and its heterocyclic bands obtained at 1250-830 $\text{cm}^{-1}$ . In halogens, fluorinated aliphatic hydrocarbons and fluorinated aromatic hydrocarbons are found in the range of

1400-1000 $\text{cm}^{-1}$  and 1300-1200 $\text{cm}^{-1}$  respectively. The P-H, alkyl phosphines, aryl phosphines, cyclic and acyclic P=N compounds shows weak to medium intensity spectra at 2500-965 $\text{cm}^{-1}$ , 2285-910 $\text{cm}^{-1}$ , 2285-1085 $\text{cm}^{-1}$  and 1500-1170 $\text{cm}^{-1}$  respectively. The aliphatic ethers and aromatic ethers of silicon compounds gives strong multiple absorption band at 1225-1060 $\text{cm}^{-1}$  and the aromatic C-O-C of ethers stretching in vibration gives strong and medium absorption band at 1270-1230 $\text{cm}^{-1}$  and 1050-1010 $\text{cm}^{-1}$  and its heterocyclic bands obtained at 1100-900 $\text{cm}^{-1}$ . The alkyl thioketones (-C=S) of sulphur compounds vibrations found at the range of 1240-1050 $\text{cm}^{-1}$  (Fig.4). The OH stretch at 3522 $\text{cm}^{-1}$  and 3400 $\text{cm}^{-1}$  confirmed the presence of gypsum and sulphate compounds. The S-O bending of gypsum observed at 669 $\text{cm}^{-1}$  and 597 $\text{cm}^{-1}$ . The S-O stretching and S-O bending of sulphates peaks appeared at 1140- 1080 $\text{cm}^{-1}$  and 680-610 $\text{cm}^{-1}$ . The P-O stretching of phosphates and Si-O stretching of Silicates spectra held at 1100-1000 $\text{cm}^{-1}$  and 1100-950 $\text{cm}^{-1}$  respectively (Palacio S *et al.*, 2014) [13]. The occurrence of  $\text{HPO}_4^{2-}$  in sulfate matrix is confirmed by the peaks of bands at 836 $\text{cm}^{-1}$  and 1015 $\text{cm}^{-1}$  (Hammam *et al.*, 2013) [7]. The sulfate group of calcium sulfate is attributed by the S-O stretching vibration at 1125 $\text{cm}^{-1}$  (Chouaya *et al.*, 2019) [5].

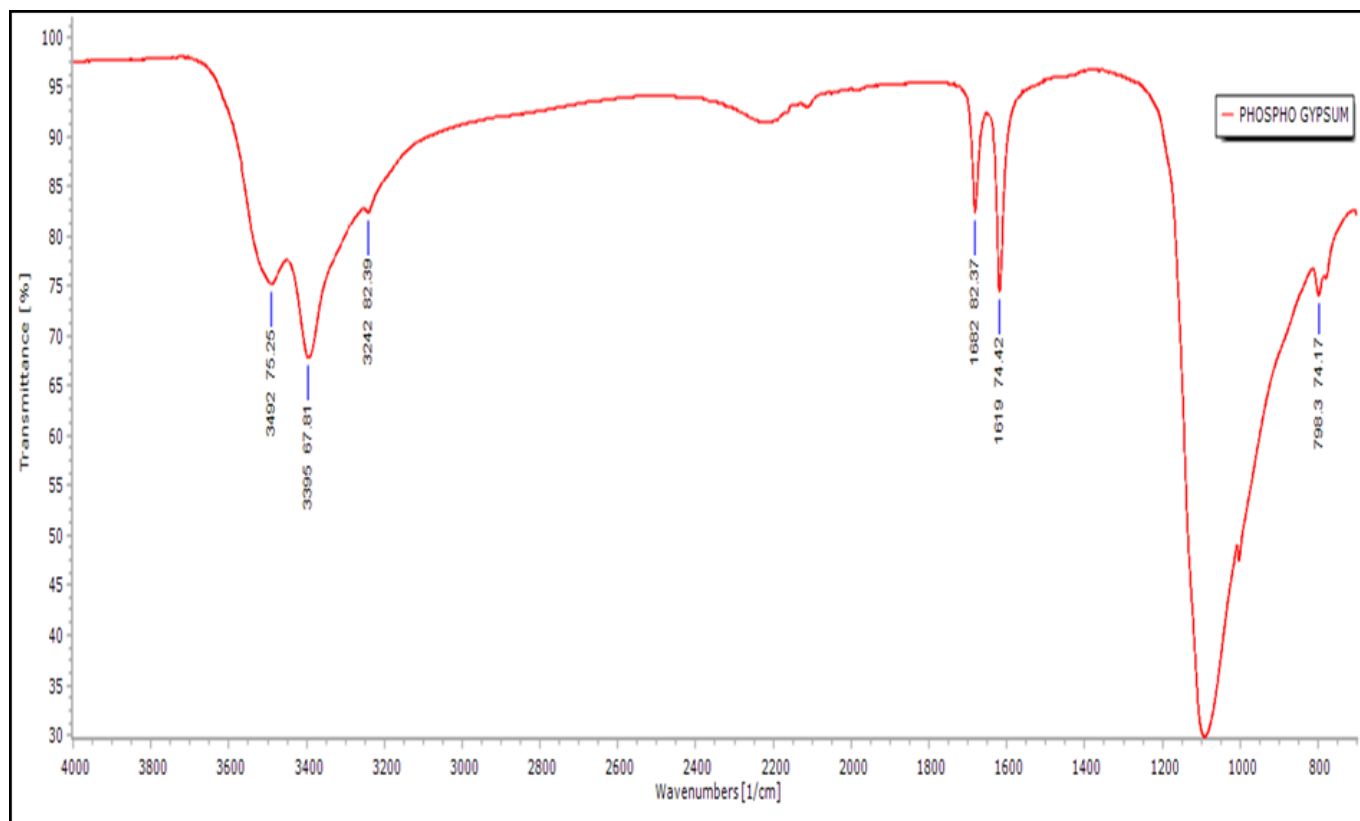
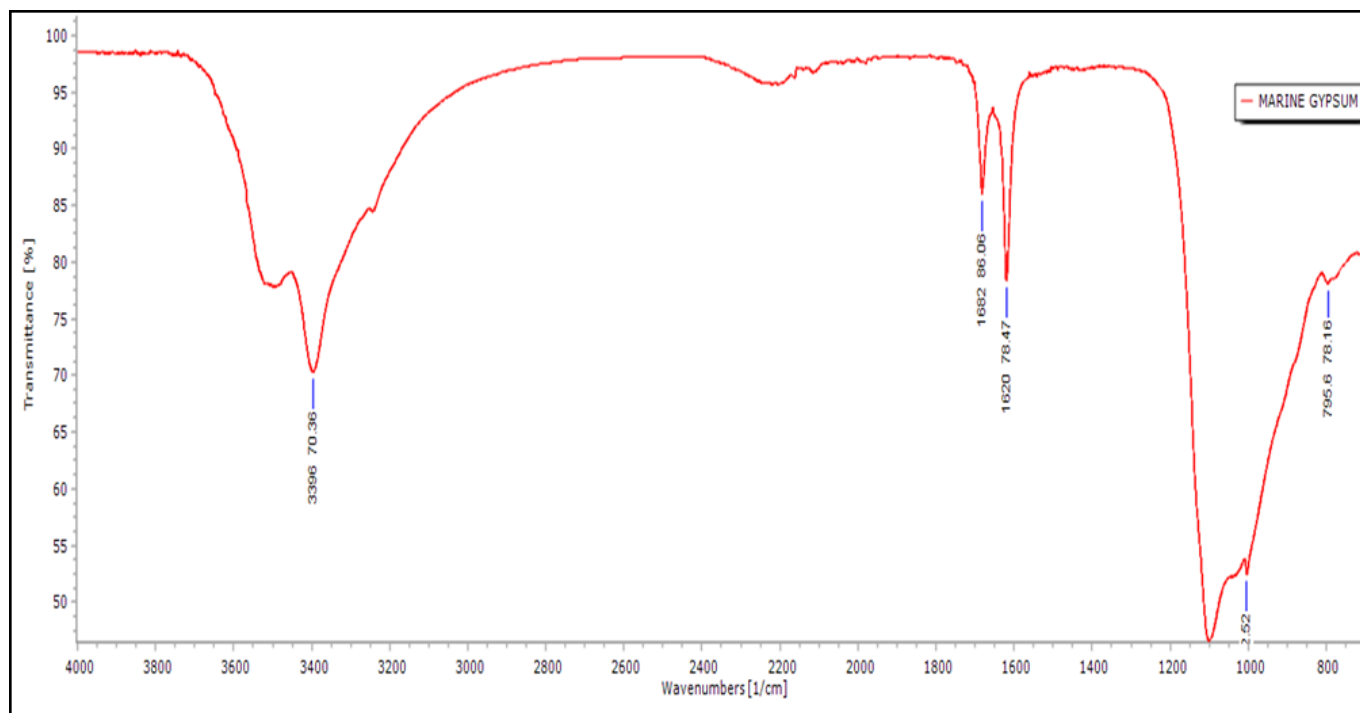


Fig 4: IR Spectra of Phospho Gypsum

#### Marine gypsum

The alcohols, nitriles, alkynes, ethers, halogens, phosphorous, silicon and sulphur compounds are identified in mineral gypsum spectral peaks. The strong intense O-H stretching of secondary and tertiary alcohols arising at  $3650-3320\text{cm}^{-1}$ , The C-OH stretching of secondary and tertiary alcohols held at range of  $1075-1000\text{cm}^{-1}$ ,  $1125-970\text{cm}^{-1}$  and  $1210-1100\text{cm}^{-1}$ . The O-H deformation vibrations found at near  $1400\text{cm}^{-1}$ . The sharp, weak to strong nitriles(C=N) stretching vibration found at  $2260-2240\text{cm}^{-1}$  which is conjugated with olefinic C=C and aromatic C=C resulting lowered frequency in absorption band and intensity of C=N absorption band. The mono substituted alkynes (RC=CH) vibrations held at range of  $3300-615\text{cm}^{-1}$  and di substituted alkynes (RC=CR') of non-conjugated stretching found at  $2260-2190\text{cm}^{-1}$ . The strong multiple absorption bands of aliphatic ethers and aromatic ethers with side chained oxygen to C atom found at  $1225-1060\text{cm}^{-1}$  and the aromatic C-O-C of ethers stretching in vibration gives strong and medium absorption band at  $1270-1230\text{cm}^{-1}$  and

$1050-1010\text{cm}^{-1}$  and its heterocyclic bands obtained at  $1250-830\text{cm}^{-1}$ . In halogens, fluorinated aliphatic hydrocarbons and fluorinated aromatic hydrocarbons are found in the range of  $1400-1000\text{cm}^{-1}$  and  $1300-1200\text{cm}^{-1}$  respectively. The P-H, alkyl phosphines, aryl phosphines, cyclic and acyclic P=N compounds shows weak to medium intensity spectra at  $2500-965\text{cm}^{-1}$ ,  $2285-910\text{cm}^{-1}$ ,  $2285-1085\text{cm}^{-1}$  and  $1500-1170\text{cm}^{-1}$  respectively. The aliphatic ethers and aromatic ethers of silicon compounds gives strong multiple absorption band at  $1225-1060\text{cm}^{-1}$  and the aromatic C-O-C of ethers stretching in vibration gives strong and medium absorption band at  $1270-1230\text{cm}^{-1}$  and  $1050-1010\text{cm}^{-1}$  and its heterocyclic bands obtained at  $1100-900\text{cm}^{-1}$ . The alkyl thioketones(-C=S) of sulphur compounds vibrations found at the range of  $1240-1050\text{cm}^{-1}$  (Fig.5). The peaks of  $3532\text{cm}^{-1}$  and  $3403\text{cm}^{-1}$  confirmed the presence of gypsum. The band assigned at  $1105\text{cm}^{-1}$  and  $668\text{cm}^{-1}$  is related to gypsum. The ranges related to sulfate stretch of gypsum observed at  $1620 - 1660\text{cm}^{-1}$  (Morillas *et al.*, 2015) <sup>[11]</sup>.



**Fig 5:** IR Spectra of Marine Gypsum

### Conclusion

In conclusion, the various functional groups such as Triazines, Azos, Silicons, Nitriles, alcohols, alkynes, Ethers, Halogens, Phosphorous and Sulfurs are identified in the sodic soil reclamation sources spectra. The peaks and bands of spectrum confirm the proportion of such compounds in individual material. The gypsum spectra found at  $668\text{cm}^{-1}$ ,  $1130\text{cm}^{-1}$ ,  $1630\text{cm}^{-1}$  and  $2200\text{cm}^{-1}$  range. The  $3522\text{cm}^{-1}$  and  $3400\text{cm}^{-1}$  ranges indicates the gypsum and sulfur compounds in sources. The S-O bending of gypsum arises at  $669\text{cm}^{-1}$  and  $597\text{cm}^{-1}$ . The peaks at  $1140\text{--}1080\text{cm}^{-1}$  and  $680\text{--}610\text{cm}^{-1}$  shows that the S-O stretching and S-O bending of sulphates. Alkyl thioketones ( $\text{-C=S}$ ) of sulphur vibrations found at the range of  $1240\text{--}1050\text{cm}^{-1}$ . This helps to confirm that the sources are suitable for sodic soil reclamation. The ranges of spectra systematically showed the presence of principle compounds such as Sulphur and gypsum materials in mentioned sodic soil reclamation sources.

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