Spectroscopy of evaporites

FARES HOWARI*

Geology Department UAE University, PO BOX 17551; Al-Ain, UAE

ABSTRACT. — The presence of salt crusts can provide information on soil salinization. Spectroscopy and remote sensing of salt crusts are useful for assessing the distribution of salt crusts in barren landscapes of arid regions as well as to evaporite accumulation in playa basins. In this study are reported the results of spectral reflectance measurements in the visible and near infrared region of gypsum, halite, calcium carbonate, sodium bicarbonate, and their mixtures. Spectral analyses have shown that the studied samples have diagnostic absorption features between 1000 to 2500 nm which are consistent with the reported values in the libraries. The positions of the absorption bands were not affected significantly quantities of evaporites per unit area. However, reflectivites increased with increasing the weight fraction of evaporite present. The reflectivity of evaporite was related to the mixing ratios in a non-linear fashion. At certain mixing ratios, the spectral reflectance data from evaporites were similar to those of the endmembers. It is interpreted that this is due to crystals coating. The reflectivity of the mixed calcium carbonate and sodium bicarbonate decrease by increasing the sodium bicarbonate fraction, due to the coating of calcium carbonate by sodium bicarbonate. The data of this study point to a possibility that the presence of gypsum, halite, calcium carbonate, sodium

bicarbonate, and their mixtures in salt crusts can be assessed quantitatively by examining the position and intensity of the absorption features. The presence of soil particles may affect these results, and this subject is currently being evaluated.

RIASSUNTO. — La presenza di croste saline può dare informazioni sulla salinizzazione dei suoli. La spettroscopia e Remote Sensing delle croste saline sono utili per stabilire la distribuzione dei sali in paesaggi privi di vegetazione di regioni aride o negli accumuli di evaporiti in bacini di playa. In questo studio si riportano i risultati di misure di riflettanza spettrale, nelle regioni del visibile e del vicino infrarosso, di gesso, halite, carbonato di calcio, bicarbonato di sodio e di loro miscele. Le analisi spettrali hanno mostrato che i campioni studiati hanno caratteri di assorbimento diagnostici tra 1000 e 2500 nm che sono coerenti con i valori riportati in letteratura. Le posizioni delle bande di assorbimento non sono influenzate in modo singificativo dalla dimensione delle particelle di halite o di gesso o dalle loro quantità per unità areale. Comunque, le riflettività aumentano con l'aumentare della frazione in peso dell'evaporite presente. La riflettività dell'evaporite è legata ai rapporti della miscela in modo non lineare. Per alcuni rapporti, i dati di riflettanza spettrale dalle evaporiti sono simili a quelli dei membri puri. Ciò viene interpretato come dovuto a Coating dei cristalli. I dati di questo studio indicano la possibilità che la presenza di halite,

* E-mail: FHowari@uaeu.ac.ae

carbonato di calcio, bicarbonato di sodio e delle loro miscele nelle croste salite possa avere valutata quantitativamente con l'esame delle posizioni ed intensità degli assorbimenti spettrali. La presenza di particelle di suolo può influenzare i risulti e questo problema è attualmente in esame.

INTRODUCTION

Salts are frequently the dominant contaminant of arid land resources. They appear on the ground surface as a result of upward capillary flow of saline water, evaporation of pounded saline water, or the exposure of geological salt deposits. Spectral patterns from these salt crusts and evaporites must be known if their distributions are to be determined remotely. Previous research indicates that evaporite minerals can be distinguished by examining adsorption features (Hunt and Salisbury, 1970; Hunt, 1982; Crowley, 1991; Csillag *et al.*, 1993; Drake, 1995; Clark, 1999). Light absorption occurs when anion groups, such as SO_4, CO_3, and water molecule are present. Since evaporite minerals contain these compounds, it is theoretically possible to identify the specific evaporite mineral through spectra matching with the known spectra libraries, provided that the evaporite consists of single evaporite mineral (Crowley, 1991; Drake, 1995; Clark, 1999).

Presently there is no clear understanding of the spectral responses of the physicochemical properties of the land surface salinity, due to the complexity of the problem (Csillag, 1993). Hence, the problem must be broken down into simpler parts and analyzed. Gypsum, halite, calcium carbonate, sodium sulphate and their mixtures are very common in the soil solutions of arid and semi-arid regions (El Ashry, *et al.* 1985; Ghassemi *et al.*, 1995; Tanji, 1996). In the act of breaking the problem down, this study will deal with the spectral characteristics of only halite, gypsum, calcium carbonate, sodium bicarbonate, and their mixtures. Weight fractions, type of mixing, and mixing ratios are the variables of major concern to this study. These variables are interlinked in a complex manner and this study will test their spectral impacts. In the present study laboratory experiments under controlled conditions were used to isolate the variables that affect the spectral responses of the surface soil salinity. The purpose is to relate reflectance spectroscopy to the chemical and physical properties of gypsum, halite, calcium carbonate, sodium bicarbonate and their mixtures, as dominant salt crusts.

This study has evolved from a series of different mental steps to understand and predict real time physico-chemical processes of salinity build up. In the first attempt satellite based multispectral mapping has been evaluated to directly investigate the salt crusts in the field, however the pixel spatial and spectral resolution were major problems that faced this effort. The second attempt was based on satellite images and focused on the indirect investigations of the salt crusts based on the physiological and morphological responses of the vegetation cover to salinity. The difficulties of the field differentiation between the responses of the vegetation cover to salinity and to other environmental factors such as light, humidity, temperature, aeration, and temporal variations were the major limitations of this line of thought. These efforts led this study to consider the reflectance spectroscopy.

The outcome from this study will test and demonstrate the potential application of multispectral data in mapping and detecting gypsum, halite, and their mixtures. In the future and combined with other data, this may lead to the delineation of areas where progressive salinization occurs, and can be used to assess the effectiveness of mitigation measures. In the future this study will also be expanded to include other evaporite mineralogies. The results from this study could lead to better understanding, and better methods for prediction of the problem based on cost effective ground and airborne based spectroscopic data.

PREVIOUS LITERATURE

During the last two decades several studies have employed remote sensing data to identify or monitor various conditions of salts and surface salinity status of soils, including the study of gypsum, halite, calcium carbonate, and sodium bicarbonate (Tuller, 1987; Price, 1990; Mougenot, *et al.*, 1993; Csillage *et al.*, 1993, Verma, 1994; Drake, 1994; Metternicht and Zinck, 1997; Dwiviedi and Sreenivas, 1998). The results have demonstrated the value of spectral techniques in efflorescences and evaporite deposit studies. However most studies and others have used digital image interpretation, image transformation and other image processing techniques in order to map the salt occurrences. Tuller (1987) reviewed the application of field remote sensing in arid land studies, and he stated that it is evident that there is need for additional careful scientific research into field remote sensing methodologies and land spectral signatures before the potential can be realized. Price (1990) concluded that four broad band spectral measurements at 930-1130 nm, 2030-2310 nm, 630-740 nm and 1600-1800 nm are sufficient to describe more than 90% of the spectral variability of the soil spectra. Mougenot and Pouget (1993) concluded that bands in the middle infrared region of the electromagnetic spectrum provide information on the differences of the salt content. Csillage *et al.*, (1993) studied the reflectance spectra of salt affected soil samples collected from California and Hungary. The study identified key spectral ranges for detecting the salts in the visible (550-770 nm), near infrared (900-1030 nm, and 1270-1570 nm) and the middle infrared (1940-2150 nm, 2150-230 nm, and 2330-2400 nm) portion of the spectrum. Drake (1995) reviewed the applications of spectroscopy in evaporate deposits. Drake used laboratory-based approach to show the effect of evaporite dehydration on the spectral responses. Several spectral catalogues are present on the World Wide Web (Salisbury, 1991; Vincent 1997; Clark, 1999) and growing volumes of studies are related to the problem at hand. Review of this literature reveals that currently there is no comprehensive spectral guide or integrated information of the spectral behavior of gypsum, halite calcium carbonate, sodium bicarbonate and their mixtures that can be used in remote sensing applications.

METHODOLOGY

The variable of major concern is the weight fraction or mixing ratios of the mixed salts. In developing the procedure to test the spectral effect of this variable, different masses of salts as well as different mixing ratios were prepared. In these experiments the salts were dissolved in sterilized distilled water (DI H_2O) in different weight fractions (10, 20, 30 and 40 grams) according to the following steps:

3.1 – The salts were dissolved in water in 1000-ml volumetric flasks;

3.2 – 200 ml of each solution was removed, placed in glass Petri dishes at 40 C° in an electric oven for 12 hours

3.3 – Immediately following the removal from the oven, the samples were placed in a desicator, after which the reflectance readings were made by means of a GER 3700 spectroradiometer. Replicate of the samples and the measurements were considered.

Most of salts encountered in soils are a mixture of two or more type of salts. Sets of chemically mixed salt samples were prepared from the 10 g/l solution fraction from which the pure salts were made using the same steps as described above. The mixing proportions are in 1 : 0, 0.75 : 0.25, 0.5 : 0.5, 0.25 : 0.75, and 0 : 1 weight mixing ratios. The spectra of the mixed samples were compared with the spectra of the pure samples of the 20 g/l solution fraction. The masses of salt (g) in the Petri dishes were divided by the area (cm^2) of the Petri dishes. All of the pure and mixed samples were examined under petroghraphic and binocular microscopes for crystal morphology observations.

In this study different approaches of data processing and digestion were considered. The traditional method of graphing the spectral data was used. Plotting the percent of reflectance versus wavelengths for the entire spectral region. Another method is the continuum removal which is of particular significance in the study of the absorption features (Clark, 1999). The continuum removal method implies the removal of the absorption features in the spectra, by plotting the intensities or band depths of the absorption features versus the associated wavelengths. This technique of spectral reconstruction can isolate the spectral features and put them on a level so that they can compared with each other (Clark, 1999).

RESULTS AND DISCUSSION:

Halite (NaCl)

Halite, sodium chloride (NaCl), is the most abundant evaporite mineral in soils (Carter, 1975; Ghasemi *et al.*, 1995). The halite samples are white to colourless, isometric or cubic crystals, with average sizes that range from 638 μm to 1850 μm (Figure 1). In most of the halite samples prepared for this study, small crystals adhere to the larger ones. About 50% to 70% of larger size crystals (2 mm) are covered with small crystal sizes that average 500 μm.

In the region of 500 nm to 1850 nm, the spectra of halite are almost featureless. In this region the spectra have a plateau shape with high reflection values that ranges from 80% to 90% (Figure 2; Table 1). Two weak absorption features were observed in this region, at 994 and 1151 nm (Figure 2). Halite has two major absorption features that appear at 1457 nm (P6) and 1958 nm (P11) (Figure 2, Table 1). Table 1 gives all the possible positions of the main absorption features of halite that might appear in the full range spectra. According to Mougenot *et al.*, 1993, halite is a transparent salt, and its chemical composition and cubic crystal system (Figure 1) do not induce strong absorption bands in the visible and near to thermal infrared. Hunt, 1972, concluded that halide ions possess no internal vibration
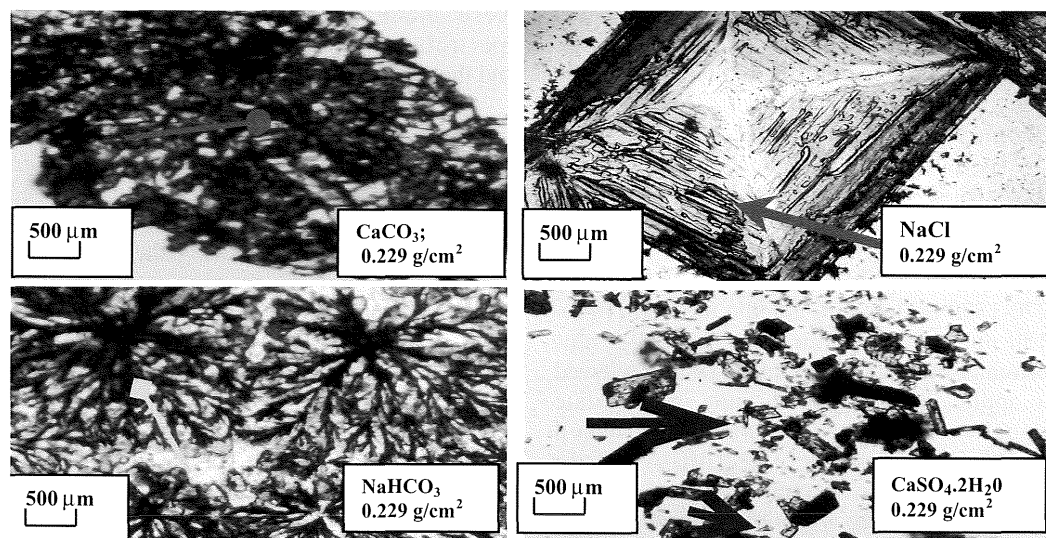

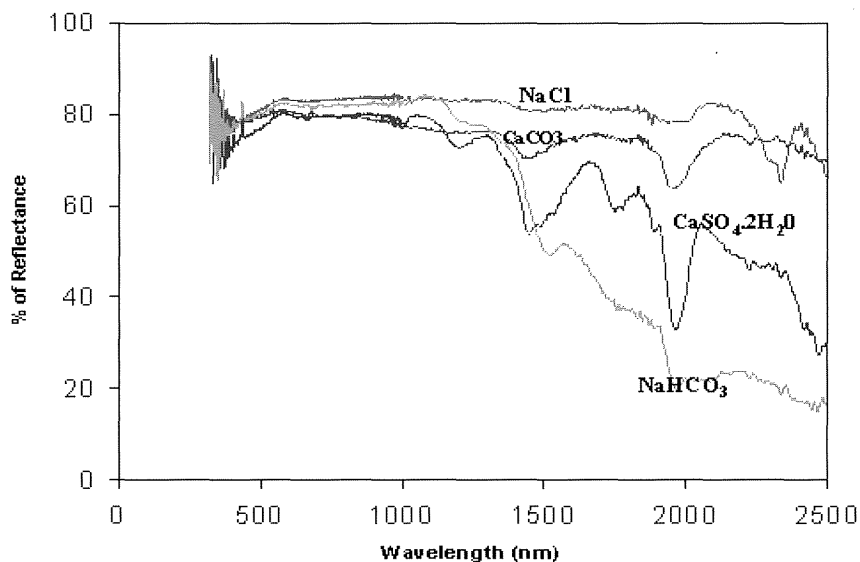Fig. 1 – Micrographs of NaCl (——▶), $CaSO_4.2H_2O$ (——≫), $CaCO_3$ (——●), and NaHCO3 (——♦)

Fig. 2 – Comparison among the spectra of NaCl, $CaSO_4.2H_2O$, $CaCO_3$ and NaHCO3.

TABLE 1

Location of the absorption features of NaCl, $CaCO_3$,
$CaSO_4.2H_2O$, $NaHCO_3$ and their mixtures.

peak name	P3	P4	P5	P6	P7	P8	P10	P11	P13	P14	P15	P16	P17
CaSO4.2HO (G)	994	1173		1435		1785		1940		2113		2310	
0.75 G: 0.25H		1124	1217	1435	1650	1795	1870	1968	2049	2183			2449
0.5 G: 0.5 H		1125	1217	1435	1650	1995		1968	2049	2183			
0.25 G: 0.5 H	1145	1217	1435	1650	1795		1968	2049	2183				
NaCl (H)		1181		1457		1795		1958			2284		
CaCO3 (Ca)			1486				1988		2123		2338	2428	
0.75 Ca: 0.25 S		1225	1489				1987		2113		2356		
0.50 Ca:	0.50 S			1225	1473			1968		2123		2356	
0.25 Ca: 0.75 S		1220	1464			1970		2126		2356			
NaHCO3 (S)	1109	1243	1498		1768		1988			2356			

modes, and the absorption features all occur in the far infrared. However, the absorption features in halite spectra are attributed to the water in fluid inclusions of halite (Hunt *et al.*, 1970, 71; Mulders, 1987; Mougenot *et al.*, 1993).

Gypsum (CaSO₄.2H₂O):

Gypsum is a hydrated calcium sulphate mineral, mostly found in nature as a prismatic, curved or twisting monoclinic crystals of vitreous luster, and as earthy, foliated or granular masses (Cole, 1974). The structure of gypsum consists of layers of SO_4^{-2} strongly bonded to Ca^{+2}. Successive layers of this type of structure are separated by sheets of H_2O molecules (Cole, 1974; Klein and Hurlbut, 1993). The gypsum crystals prepared here have fine particle size; the average crystal size was 470 μm (Figure 1). The samples are white to colorless with monoclinic crystals. In gypsum the overtones or combination tones from fundamental vibrations of the water molecules produced a series of bands affecting the infrared spectrum between 1000 and 2500 nm, e.g. 1464, 1550, 1750, 1978 and 2300 nm, and can be detected in the spectra of gypsum.

Calcium carbonate (CaCO₃):

Calcium carbonate is one of the most common minerals on the face of the Earth. It comprises about 4% by weight of the Earth's crust and is formed in many different geological environments. Of major concern to this study is the accumulation of calcium carbonate at or near the surface of the soil which is very common on the arid and semi-arid environment.

The prepared calcium carbonate crystals of this study were white with very fine aggregate of crystals; their average crystal size is 400 μm (Figure 1). The spectra of calcium carbonate usually has several absorption features at wavelength in excess of 1498 (P6) due to vibrations of the carbonate radical; these absorption features are situated near 1763 (P8), 1876, 1990 (P8), 2167, 2333 (P13) and 2500 nm (P17) (Gaffery, 1989, Drake, 1995, Clark, 1999). The observed spectrum of calcium carbonate of this study displays carbonate bands in the range of at 1988 nm (P11), 2123 nm (P13), and 2338 nm (P15), and 2428 (P17) (Figure 2 and Table 1). The spectral profiles of calcium carbonate shows the highest reflection

values occur between P13 and P15. These absorption features are due to the vibrational modes of CO_3^{-2} ion.

Nahcolite (NaHCO₃):

Nahcolite (NaHCO₃), is a naturally occurring sodium bicarbonate. Its structure consists of planar chains of carbonate groups linked with hydrogen bonds; planes are linked together by sodium in six-fold coordination with oxygen (Klein and Hurlbut, 1993). $NaHCO_3$ is also known chemically as «baking soda». In this study the sodium bicarbonate samples have white, very fine rosy crystals that range from 290 μm to 1100 μm with an average crystal size of 850 μm (Figure 1). The spectra of sodium bicarbonate exhibit a large diagnostic slope from 1243 nm (P5) to 2356 nm (P15). The spectrum of sodium bicarbonate shows the higher reflection value around the 1109 nm (P4) (Fig. 3). Several absorption features can be seen in the spectra of sodium bicarbonate. These absorption features appear at 1243 (P5), 1498 (P6), 1790 (P8), 1988 (P11) and 2356 nm (P15), among the salts, sodium bicarbonate has the most diagnostic spectral pattern (Figure 2).

Mixture analyses or weight fraction:

NaCl (H) - CaSO₄.2H₂0 (G): This figure shows that the spectra of the chemically mixed samples are different from the pure spectra of gypsum and halite. Most of the peak intensities, especially P4, P10, P14, and P16, vary according to the mixing ratios. Using the continuum removal method, this variation is expressed graphically in Figure 5. The peak intensity of the mixed samples decreases by decreasing the gypsum fraction, and 80% of the investigated peaks follow this pattern. A non-linear relation was found between the reflectivity of the chemically mixed gypsum and halite, and the mass fractions or the mixing ratios of the samples (Figure 3 and 4). When the mass fraction of gypsum decreases, the spectral contribution of halite eclipses the spectral features of the gypsum. Lindberg and Smith 1973, indicated that when gypsum is
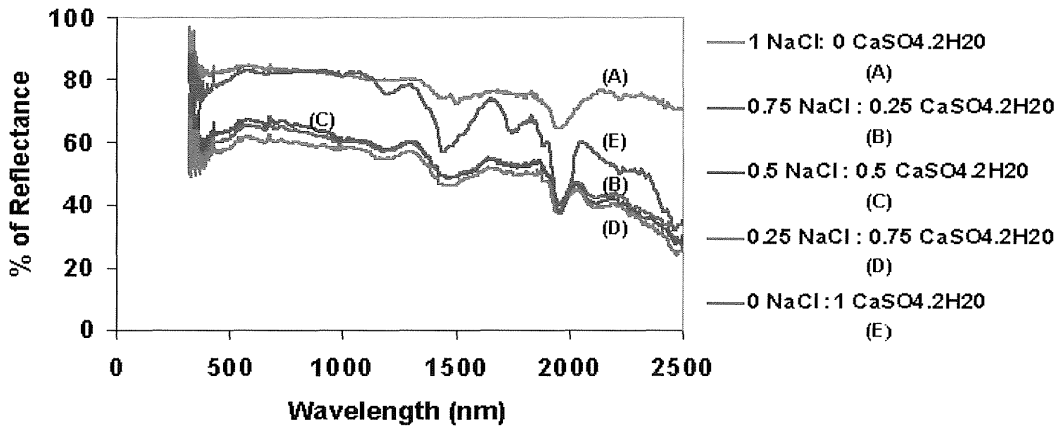
Fig. 3 – Comparison among the spectra of NaCl, CaSO$_4$.2H$_2$O and their chemical mixtures.
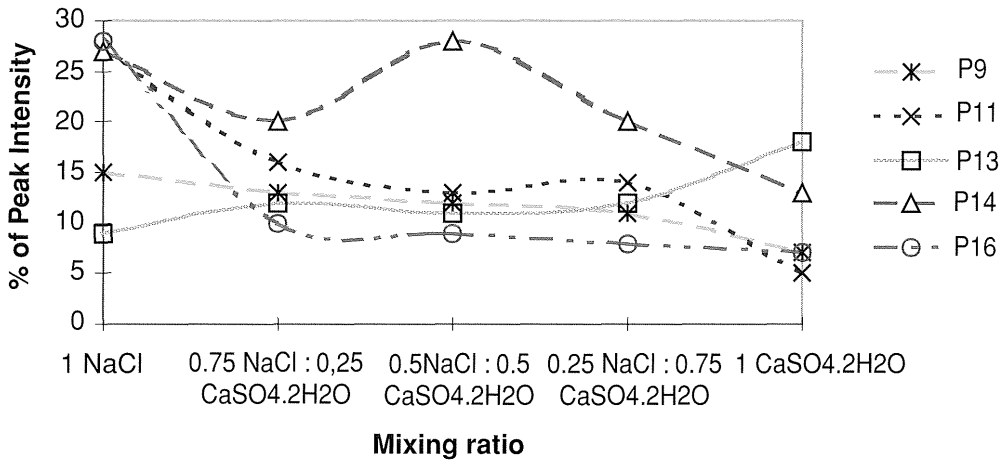

Fig. 4 – Behavior of the peak intensity of selected absorpiotn features of gypsum and halite versus chemical mixing ratios; using the continuum revmoval method

mixed with other evaporite salts, some bands disappear and the intensity and sharpness of the strong peaks decrease. This could be attributed to the spectral interferences between gypsum and halite in the mixed samples and/ or the coating of one salt over the other.

By inspecting the mixed (MS) and pure samples (PS) under the optical and plain microscopes, crystal overgrowth was observed in the mixed samples. This could be due to the fact that the nucleation and the growth rate of the PS and MS are different from each other. The first is homogeneous, and the second is relatively a heterogeneous system. This causes

differences between the sizes and habits of the crystals among the PS and MS. Gypsum has white milky color, and halite has glassy transparent color. The colors of the mixed samples are different from the colors of gypsum and halite, they are less transparent. Photons have a high probability of encountering less transparent grains (Clark 1999). According to Clark, 1999, this can drastically reduce the reflectance values much more than the weight fraction and less than or equal to the effects due to grain size. This result in lower reflectivity of the spectra of the mixed samples. The spectral interference between gypsum and halite crystals in the mixed samples are another cause of their lower reflectivities. The causes and effects of the spectral interference is discussed in detail in Gupta, 1991; Vincent, 1997; Gaffey, 1989; Asrar, 1989 and others.

Covering of one salt by another is controlled by the solubility product of the salts, based on the principal, the first to precipitate the last to dissolve (Tanji, 1996). The solubility of the NaCl (6108 meq/ litter) is higher than the solubility of $CaSO_4.2H_2O$ (30 meq/l) and under certain conditions is expected that after evaporation of the 81%, $CaSO_4.2H_2O$ will be completely precipitated; then after evaporation of about 90.5% of the water, NaCl is precipitated. Therefore, halite will coat gypsum and prevent the detection of gypsum spectra if it has sufficient amount and thickness to cover the gypsum and fill the field of the view of the spectroradiometer.

$CaCO_3$ (C) - $NaHCO_3$ (S): In the range of 400 nm to 1450 nm, the mixed samples of $CaCO_3$ and $NaHCO_3$ reveals reflection values less than the reflection values of those of 1) calcium carbonate spectra, and 2) sodium bicarbonate spectra (Figure 5). This figure depict that in the range of 1464 (P6) to 2428 (P17) nm, the mixed samples has a reflection values in between those of the endmembers, calcium carbonate and sodium bicarbonate (Figure 5 and Table 1).

In the range of 450 to 2500 nm, the reflection

values of the mixed samples are less than those of carbonate spectra and higher than those of the sodium bicarbonate spectra. The study investigated the relation between the mixing ratio and the reflectiviy, it was found that the mixing ratio affects the detected spectra of the mixed samples in a nonlinear fashion (Figure 5) due to multiple scattering.

EXPECTED APPLICATION

The results reported here can be used to (1) identify and map selected salt crusts soil surfaces, and (2) evaporite accumulations of the playas. According to Tanji, 1996 and Drake, 1995, the presence of particular diagnostic minerals on the playa or soil surfaces give an indicator of those solutes that are common in the brine from which these evaporite or salts have been derived from. Gypsum is diagnostic mineral for $Na-Ca-SO_4$-Cl and $Mg-Na-Ca-SO_4$-Cl brines, and nahcolite is diagnostic for $Na-CO_3-SO_4$-Cl. Therefore this study adds a link between spectroscopy and the evaporite and/ or salt crusts evolution.

CONCLUSIONS

The features in the studied spectra are sufficiently characteristic to be used for analytical purposes. Although any two pure or mixed gypsum and halite spectra may well have one or more of their absorption features in a similar position, the observed variations in the position, spacing, size and shape of these features means that their spectra can be diagnostic. Thus, the spectral differences of the salts appear to be significant to determine the identity and characteristics of gypsum, halite and their mixtures and/ or calcium carbonate and sodium bicarbonate and their mixtures. The positions of the absorption bands were not affected significantly by their quantities per unit area. However, the reflectivity increased with increasing the amount or the concentration of the salts. In most cases halite spectral pattern
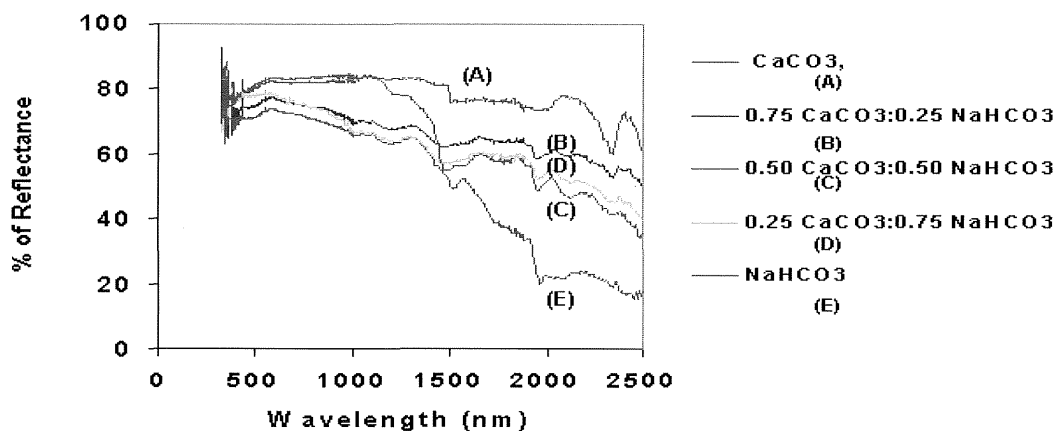
Fig. 5 – Comparison among the spectra of CaCO₃, NaHCO3 and their chemical mixtures.

eclipses the spectral features of the gypsum in a nonlinear fashion, when the mass fraction of gypsum is reduced to about 75% in mixed salt evaporites.

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