

Gypsum Formation in Sulphur Treated Soils

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ABSTRACT. Soil column experiments were conducted to evaluate the effect of different rates of sulphur application on gypsum formation in calcareous soils. Elemental sulphur was applied to three soils at the rate of 0.5%, 1.5% and 3.0%. Soils columns were incubated at 30°C for 3,6,9 and 18 weeks and were intermittently leached with distilled water. Thin soil sections were microscopically examined to detect and evaluate gypsum formation in soils. Chemical analysis of gypsum was also used for the same purpose. Gypsum, as anisotropic anhydrous aggregates, was noticed with thin sections in all sulphur treated soils. Formation of gypsum was confined to the uppermost 10 cm and was optically recorded after 9 and 18 weeks of incubation treatments for two of the Soils, of Al-Qasseem and Derab at all periods of incubation for the Al-Kharj sandy soil. These findings were in agreement with the chemical data which showed the same trend. The second level of sulphur (1.5%) application is highly recommended as most of it can be oxidized after (9-18 weeks).

Application of sulphur, as a soil amendment, is a common Practice. Under ideal conditions, sulphur is converted into sulphuric acid within a few weeks by microorganism, (Li and Caldwell 1966). The sulphuric acid then acts to reduce the relatively high pH values of these soils and to dissolve native lime (Mee *et al.* 1985). In turn, released Ca would replace the sodium on the exchange complexes and also react with sulphate produced to form gypsum. Accordingly, gypsum formation in these soils is largely controlled by the speed in which the oxidation mechanism is operating. Therefore, determination of gypsum could well fairly estimate the rate of sulphur oxidation in soils.

The majority of soils in Saudi Arabia are calcareous with alkaline pH. On the other hand, Saudi Arabia produces about 1.4 million tons of elemental sulphur annually (Sadiq 1985). So, elemental sulphur is available and less costly material for amending soils of Saudi Arabia.

Extraction and determination of soil gypsum either by gravimetric, potentiometric or turbidimetric methods is difficult and time consuming (page *et al.* 1982). X-ray diffraction was suggested for detecting gypsum in soils by (Brindley and Brown, 1980). However, this technique could not be of much use with few and poorly crystalline gypsum aggregates. Optical examination, through microscopic observation of thin soil sections was also reported to be useful for identification and estimation of gypsum in soils by (Brewer 1976). Also, microscopic studies of thin soil sections could produce additional information such as grain size, degree of crystallinity, distribution and mode of occurrence of gypsum. Because of these numerous advantages, this latter technique was chosen to be used in the present work.

The main objective of the present work was to detect, through microscopic observations, the type and rate of changes which occur in calcareous soils amended with elemental sulphur. The changes, as expressed by the formation of gypsum, were evaluated under different sulphur application rates and incubation periods.

Materials and Methods

Three surface soil samples (0-30 cm) from the agriculturally important areas in Saudi Arabia, Al-Qasseem (Torrifluvent), Al-Kharj (Torripsamment), and Derab (Torrifluvent) were used for the current work. Soil samples were air-dried and crushed to pass through a 2 mm-sieve, thoroughly mixed, and placed in 45 cm long glass columns having an inside diameter of 4.5 cm. Elemental sulphur (< 75 micron) was mixed with the top 10 cm of the soil in each column at a rate of 0, 0.5, 1.5, and 3.0%. Distilled water was added to bring the soil to 130% of the field capacity at the beginning of the experiment, and water (30% of the field capacity) was added every 10 days. Soil columns were kept in constant temperature chamber at 30°C for 18 weeks. After periods of 3, 6, 9 and 18 weeks, the soil columns were sectioned into five segments, *i.e.* 0-5, 5-10, 10-20, 20-30, and 30-40 cm. Undisturbed samples from each segment were taken for micromorphological studies, whereas the rest was used for chemical analysis.

Gypsum was determined in both sulphur treated soils as well as the control, using the precipitation method with acetone (Richards 1954). The untreated soil samples were analyzed for pH, electrical conductivity in the saturation extraction (Richards 1954). Particle size distribution was carried out by the pipette method after (Kilmer and Alexander 1949). Organic matter was measured according to the Walkey-Black method, (Black 1965), calcium carbonate was determined volumetrically using Collin's Calcimeter, (Williams 1948).

Epoxy resin was used, under vacuum, for soil impregnation and preparation of thin soil sections followed those techniques proposed by (Brewer 1976). Instead of water Kerosine was used for grinding and polishing to avoid dissolution of gypsum. The prepared slides were studied using a polarizing microscope.

Results and Discussion

Soils under investigation varied in carbonate content, soluble salts and, texture (Table 1). Elemental sulphur(s), under plane polarized light in thin sections, appears as yellow aggregates (Figs. 1&2a). Due to its isotropic nature; (Fig. 2b), it appeared completely dark under crossed nicols. On the other hand, gypsum (g) is an anisotropic mineral which is colorless with an anhedral to subhedral form and has a very week birefringence. Based on measurement of areas occupied by sulphur relative to those replaced by gypsum aggregates in thin-soil sections, the rate of sulphur oxidation, as expressed by the formation of gypsum, was estimated.

Inspection of various thin soil sections of the uppermost 10 cm samples for the first rate of sulphur application indicated that sulphur was rarely detected in

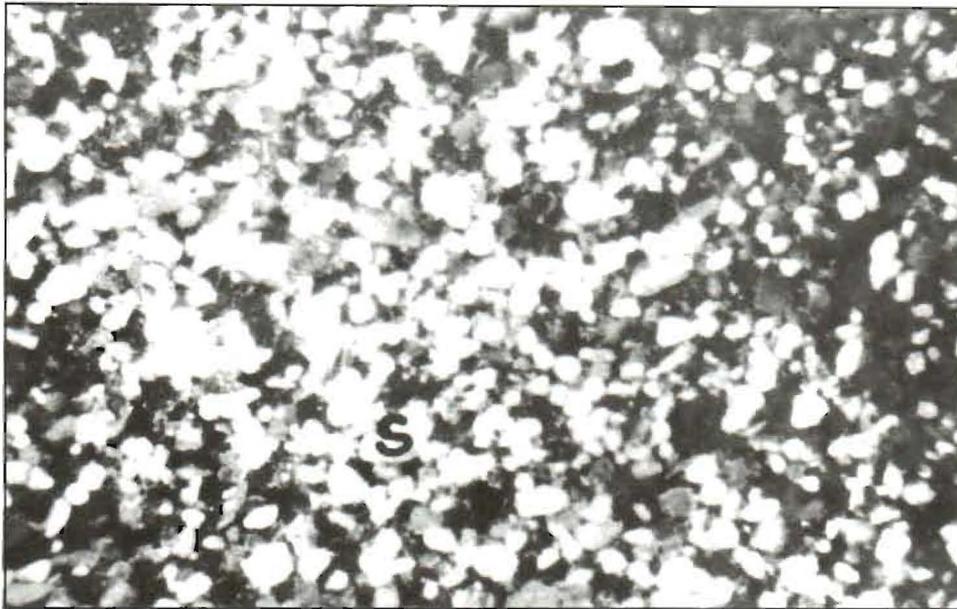


Fig. 1. Section showing the elemental sulphur between the soil materials. Thin section in plane light. X 60. Soil of Al-Qasseem, third rate of sulphur and after 3 weeks.

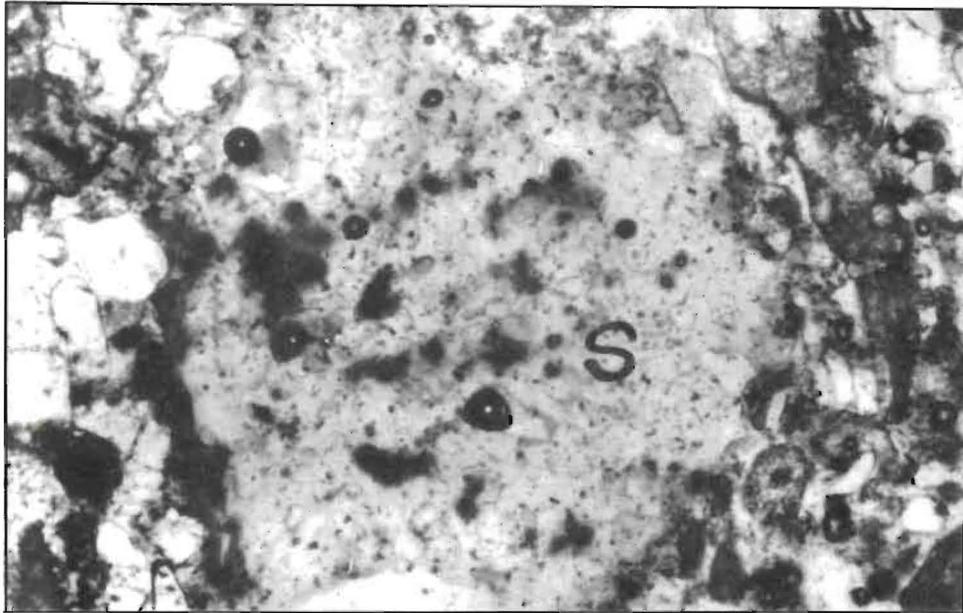


Fig. 2a. An aggregates of sulphur, partially oxidized at the edges. Derab soil at the third rate of sulphur and 18 weeks. Thin section in plain light. X 250.

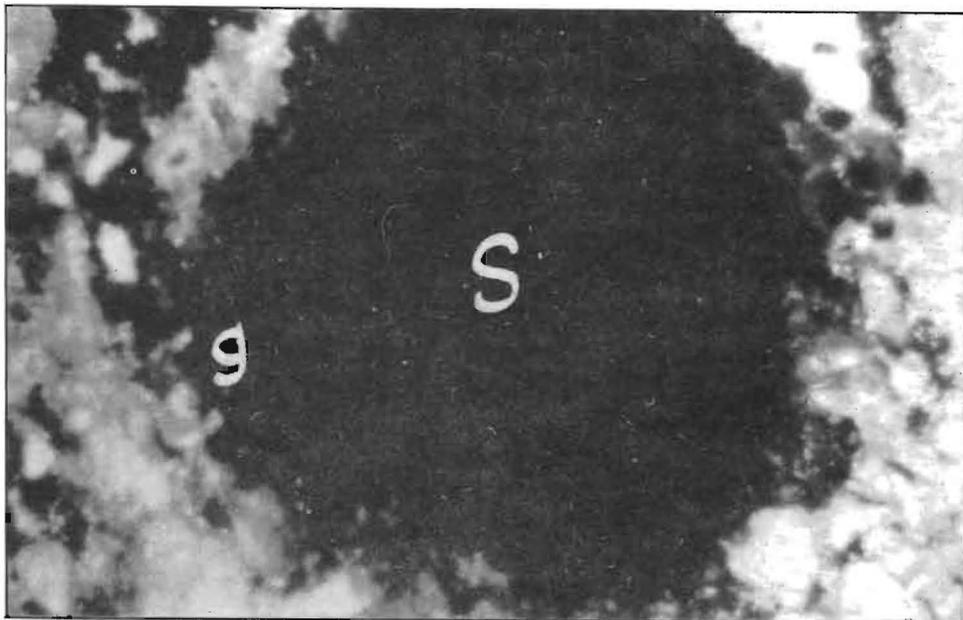


Fig. 2b. Thin gypsum merritic formed the edges of sulphur aggregate (dark under crossed polarizers). X 250. g = gypsum

Table 1. General characteristics of the soils

Location	Soils	pH	E.Ce ms/cm	Carbonate %	Gypsum %	Organic matter %	Mechanical analysis texture			
							Sand %	Silt %	Clay %	
Al-Kharj	(Torripsamment)	8.5	2.2	21.1	-	0.23	88.3	6.0	5.7	Sandy
Al-Qasseem	(Torrifluvent)	8.4	3.1	6.6	-	0.15	78.8	11.4	9.8	Sandy loam
Derab	(Torrifluvent)	8.8	0.4	42.4	-	0.08	82.8	10.2	7.0	Loamy sand

those cases following 9 weeks of equilibration. Most sulphur (80-90%) was oxidized, and numerous gypsum aggregates resulted, (Fig. 3). After 18 weeks of incubation, almost all (90-100%) of added sulphur was oxidized with the second level of sulphur application. Slightly lower rate of sulphur oxidation (70-75%) occurred after 9 weeks of equilibration for both, Derab and Al-Qasseem soil samples. A much lower oxidation rate (about 50%) resulted in Al-Kharj soil. This could possibly be the result of relatively limited activity and/or low population of sulphur oxidizing microbes in the sandy soil of Al-Kharj. Almost equal sulphur oxidation rates, however, were reached (75-90%) in all cases 18 weeks after sulphur application. For the third rate, most of the added sulphur (about 70%) was not oxidized after 9 weeks. Increasing the period to 18 weeks did not markedly increase sulphur oxidation, as it reached only 40-50% of the added sulphur in all three soils under study. These data clearly show that, in all cases, the percentage of oxidized sulphur decreases as the rate of application increases. This is in agreement with some other studies carried out on similar soils (Shaddfan and Hussen 1985) and (McCready and Krouse 1982).

Gypsum, as fine anhedral aggregates (micrites), was optically detected in all sulphur-treated soils which mainly occupied the uppermost 10 cm of soil columns.

Large amount of soluble sulphates were considerably present in the leachates of sulphur treated soil columns. This indicates the downward movement of dissolved sulphates resulting from sulphur oxidation. High moisture level or continuous wetness of soil material below the surface 10 cm depth may be unfavourable for the formation of gypsum, as sulphates were mainly kept in the soluble form.

The area of gypsum aggregates in the first and second sulphur application rates ranged between 3 and 5% of the total area of thin section and reached 6-8% in the third rate of application; sulphur granules of $< 100\mu$ in diameter were noticed to be completely oxidized. This was mostly the case with the first and second application rates, where gypsum was formed after elemental sulphur, (Fig. 4). In samples receiving the highest rate of sulphur, larger granules (patches) with a diameter up to 1mm of unchanged sulphur were frequent. Oxidation of such coarsely aggregated sulphur was not evident (Fig. 5). Few and thin micritic gypsum formations were only present at the surfaces of sulphur granules that were mostly coated with fine soil material (Fig. 2b). The low specific surface of coarse sulphur particles was possibly caused the low oxidation rate of sulphur that was obtained. The same behaviour was reported by Li and Caldwell (1966) who found that sulphur oxidation in sandy loam soils was increased with a decrease in particle size and most oxidation occurred with particle size below 100 mesh (150μ).

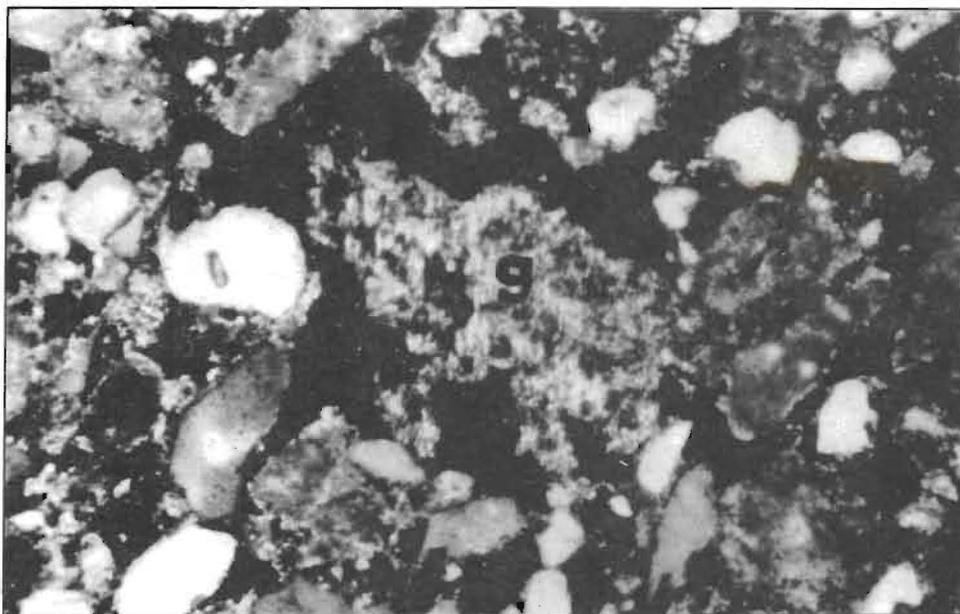


Fig. 3. Numerous gypsum aggregates (gray colour), Qasseem soil, first sulphur rate after 9 weeks. Thin section under crossed polarizers. X 250. g = gypsum

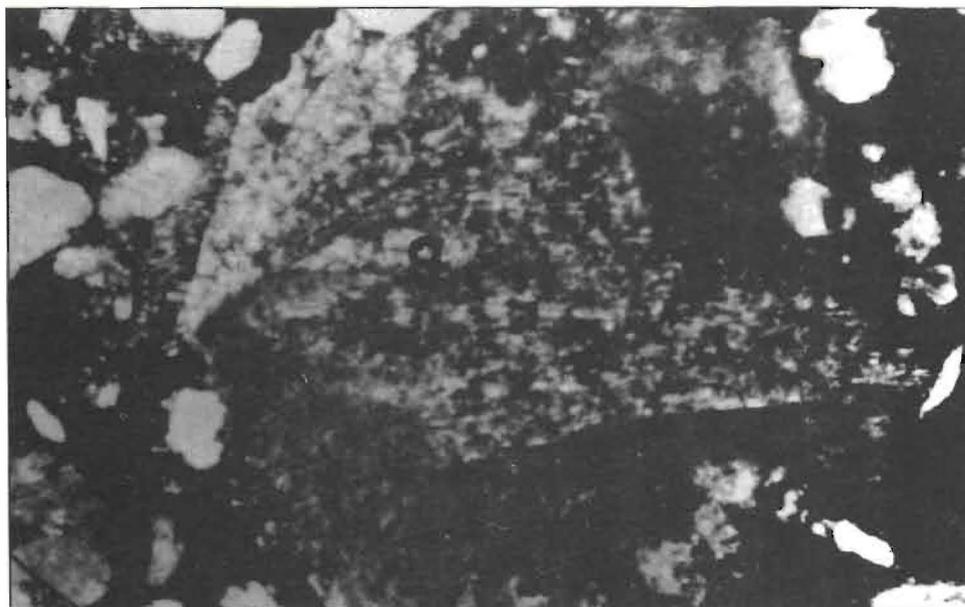


Fig. 4. Gypsum aggregates (gray colour) coated with fine soil materials. Al-Kharj soil, second rate of sulphur, after 9 weeks. Section under crossed polarizers. X 250. g = gypsum

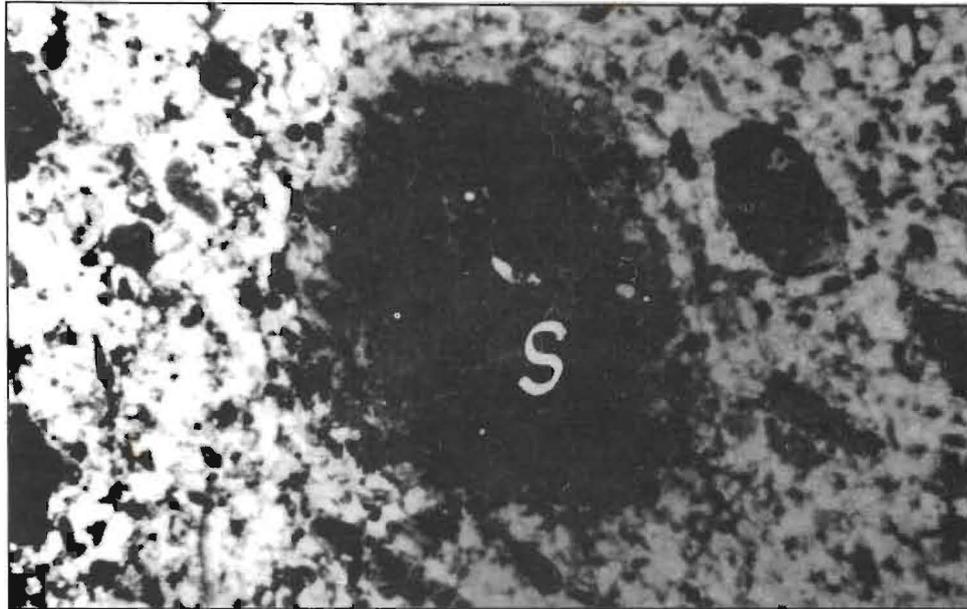


Fig. 5. Partially oxidized sulphur, Derab soil, third rate of sulphur, 18 weeks. Section in Plain light. X 60.

It is clear from (Fig. 6 & 7) that gypsum was chemically detected at all rates of sulphur applications. In all cases, an increase in gypsum content with increasing of sulphur application and time of incubation was evident. These results are in agreement with the microscopic observations. Similar trends were found by Shaddfan and Hussen (1985) who reported that high rates of sulphur significantly increased the electrical conductivity and the gypsum content of soil. Fig. 6 & 7 show that gypsum was detected after 9 weeks incubation for both, Al-Qasseem and Derab soils.

While in Al-Kharj soil, gypsum was chemically detected at all periods of incubation. Al-Kharj soil is of relatively coarse texture as compared to the Derab and Al-Qasseem soils and dessication with subsequent precipitation of gypsum is expected to be more frequent in Al-Kharj soil. McCready and Krouse (1982) mentioned oxidation being most effective between 30°C and 40°C. Accordingly, a high oxidation rate is expected in soils of Saudi Arabia due to the favourable temperature, especially if the soil is not highly saline and of suitable moisture content.

Abd-El Fattah and Hilal (1985) reported that the level of 0.5% sulphur for sandy soil, the level of 1% sulphur for alkaline clay loam soil, and 3% for

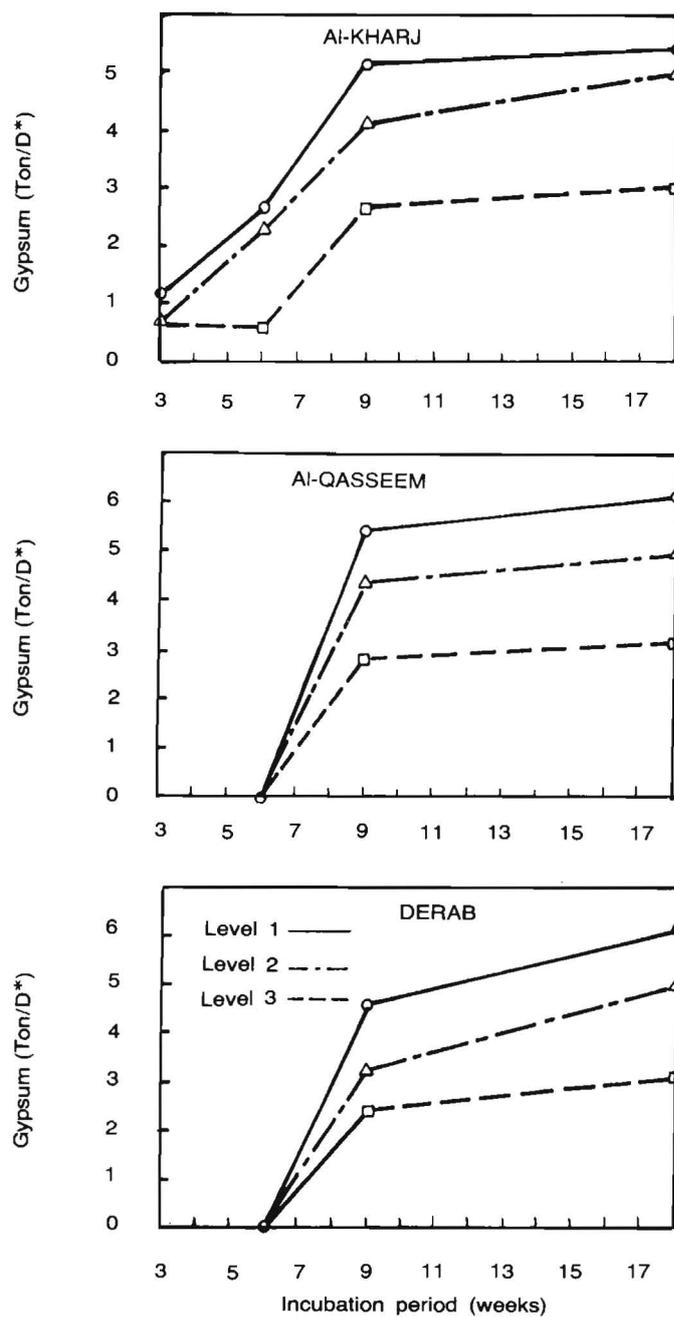


Fig. 6. The amount of gypsum formed in soils (0-5 cm) at different levels of sulphur.
* D = 1000m².

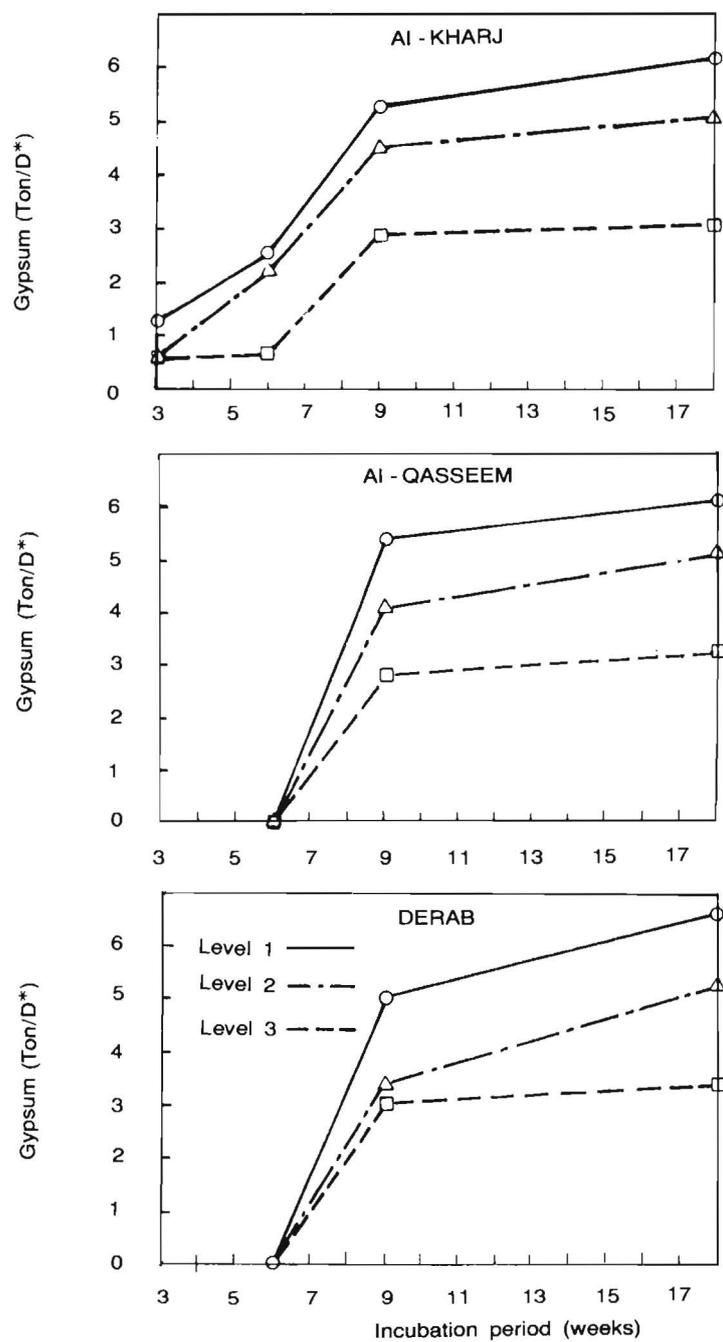


Fig. 7. The amount of gypsum formed in soils (5-10 cm) at different levels of sulphur.
* D = 1000m².

calcareous soil may be applied without harmful effect. The second level of sulphur application is highly recommended as most of it can be oxidized after a relatively reasonable time (9-18 weeks). Accordingly sulphur application, to be most efficient in reclaiming the soil under study, may be within the second level of sulphur addition, *i.e.*, 1.5%.

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تكوين الجبس ببعض الترب السعودية المعاملة بالكبريت

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أجريت الدراسة لتقييم تأثير اضافة الكبريت على تكوين الجبس ببعض الترب الصحراوية الجيرية. أضيف الكبريت لثلاثة أنواع من ترب المنطقة الوسطى بالمملكة العربية السعودية. ولقد أضيف الكبريت بمعدلات مختلفة:

٠,٥ ٪، ١,٥ ٪، ٣ ٪. ولقد تم خلط الكبريت المضاف للطبقة السطحية بأعمدة التربة المختلفة والتي حضنت على درجة ٣٠ مئوية لفترات مختلفة:

٣ أسابيع، ٦ أسابيع، ٩ أسابيع، ١٨ أسبوعاً، ولقد فحصت مقاطع التربة الرقيقة مجهرياً للتعرف على توزيع وكمية كل من الكبريت والجبس بالترب المختلفة. وقد تم تقدير الجبس كيمياوياً بالاضافة إلى تقديره مجهرياً.

أوضحت الدراسة المجهرية لمقاطع التربة الرقيقة تكون الجبس على هيئة تجمعات في جميع الترب المعاملة بالكبريت. يتميز الكبريت تحت المجهر بألوانه الصفراء في الضوء المستوي ويكون مقسم تحت الضوء المستقطب المتعامد.

لوحظ من الدراسة المجهرية تكون الجبس بالطبقة السطحية فقط (صفر - ١٠) ولم يستدل على أي تكوينات جبس في طبقات التربة، ولقد بدأ الجبس في التكون بعد فترة تحضين ٩ أسابيع في تربة كل من القصيم وديراب وبعد ٣ أسابيع بتربة الخرج الرملية وذلك عند جميع مستويات الكبريت المستخدمة.

ولقد لوحظ زيادة كمية الجبس المتكونة بزيادة كمية الكبريت المضافة للتربة.

ولقد أوضحت الدراسة أن النتائج المتحصل عليها من الفحص المجهرى والتحليل الكيماوي للجبس كانت متوافقة في كثير من المجالات .
ويمكننا القول بناء على نتائج الدراسة أن المستوى الثانى من الكبريت قد يكون أفضل المستويات المستخدمة ولكن هذا يحتاج إلى دراسات مكثفة .