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STUDY OF CARBONATE ROCKS OF THE KHUFF AND JUBAILA FORMATIONS , SAUDI ARABIA BY THERMAL DERIVATOGRAPHY

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دراسة صخور الكربونات لمتكوني الخف والجوبيلا بالمملكة العربية السعودية بالتحليل الحراري الديريفاتوجرافي

اظهر التحليل الحراري الديريفاتوجرافي ان صخور كربونات متكوني الخف والجوبيلا تتكون من الكالسيت او الدولوميت (من النوع الكالسيتي) او كلاهما واللذان تم دراسة سلوكهما الحراري التفاضلي والوزني باستفاضة . وقد وجد ان للمكونات النسيجية لصخور الكربونات مثل الميكريت Micrite، الاسباريت Sparite، ومكونات الـ Allochems بعض التأثير على حدة الـ Peaks ودرجة حرارة التفاعلات المختلفة . وقد استخدمت نتائج التحليل الحراري الوزني في التقدير الكمي للكالسيت والدولوميت منفردين أو مجتمعين وكانت النتائج متوافقة مع نتائج التحليل بحيود الأشعة السينية، التحليل الكيميائي مما يؤكد نجاح وفاعلية الطريقة المستخدمة في هذا البحث .

Key ward : Carbonate rocks, khuff and jubaila, Saudi Arabia , thermal derivatography

ABSTRACT

Thermal derivatographic analysis of the Khuff and Jubaila carbonate rocks shows that calcite and / or dolomite (of calcitic type) are the main constituent minerals. Their differential thermal and thermogravimetric behaviours are thoroughly investigated . Textural rock components (sparite , micrite and allochems) were found to have some effect on the sharpness of peaks and the temperatures at which the reactions take place. Thermogravimetric data are used for the quantitative estimation of calcite and dolomite. The results show very close similarity to those obtained by X-ray diffraction and chemical analyses.

INTRODUCTION

Thermal methods of analysis are most commonly applied for the mineralogical and petrographic investigations of minerals and rocks such as clays, carbonates , hydrated sulphates, salt minerals and coal, etc.

Heating of minerals causes chemical and physical transformations to occur. The transformations are accompanied by the absorption or liberation of heat (endothermic or exothermic reactions , respectively). Those thermal effects can be detected with a high degree of sensitivity by the differential thermal analysis (DTA) technique. Furthermore, the above reactions also involve changes in weight which, in turn, can be determined with a high accuracy by the thermogravimetry (TG) technique . The total course of thermal transformations occurring in the sample is indicated by a third curve, differential thermogravimetry (DTG) curve which is very useful in distinguishing reactions which occur in a close time sequence and merged together in the ordinary TG curve.

The "derivatograph" is an instrument capable of simultaneously recording the DTA, TG and DTG curves and permits the exact interpretation of thermal effects. Such interpretations are very useful not only for identification of minerals but also for their quantitative estimation. Among authors who utilized this instrument for quantitative estimation of minerals are Selmeczi [1], Langier Kuzniarowa [2], El Askary [3] and Stankowska [4]. Their quantitative estimations were based on the fact that the amount of the mineral can be drawn if its reactions are not influenced by other minerals present in the rock sample, i.e., if their DTA peaks do not overlap or at least if the DTG curve reveals such overlapping.

In the present work the derivatograph is used for the study of the Khuff and Jubaila carbonate rocks to fulfil the following objectives:

- Identification of the constituent minerals and interpretation of their differential thermal and thermogravimetric behaviours.

- Investigation of the effect of textural components, i.e., orthochems and allochems on such behaviour.

- Quantitative estimation of constituent minerals and comparing the results with those obtained from X-ray diffraction and chemical analyses.

GEOLOGY AND PREVIOUS WORK

The Late Permian Khuff and Late Jurassic Jubaila formations crop out along two belts in a N-S trend in Central Saudi Arabia. These belts are subparallel to the eastern margin of the Precambrian basement complex of the Arabian Shield which occupies western Arabia. Powers et al. [5] and Powers [6] mentioned that the Khuff and Jubaila formations can be traced along strike for a distance of about 1200 kms and 1100 kms, respectively Fig.1. They also mentioned that the Khuff Formation is composed of limestones and dolomites alternating with red and green gypsiferous shale while the Jubaila Formation is made up of partially dolomitized aphanitic limestone with occasional beds of pellet skeletal calcarenitic limestone and tightly cemented calcarenite.

The stratigraphy, lithology and facies of the Khuff and Jubaila formations are extensively studied by some investigators, among them are : Powers et al. [5], Powers [6], Zeidan [7], Basyoni [8& 9] and Al Laboun [10].

Recently, Basyoni et al. [11] carried out comprehensive petrographic and geochemical studies for the Khuff and Jubaila carbonate rocks. They also studied the economic potentiality of these rocks as reservoirs for hydrocarbons and as raw materials in several industrial applications. Banat et al. (L127) showed that the Ca: Mg stoichiometry and Sr content differ in Khuff and Jubaila dolomites due to the difference in their depositional environments.

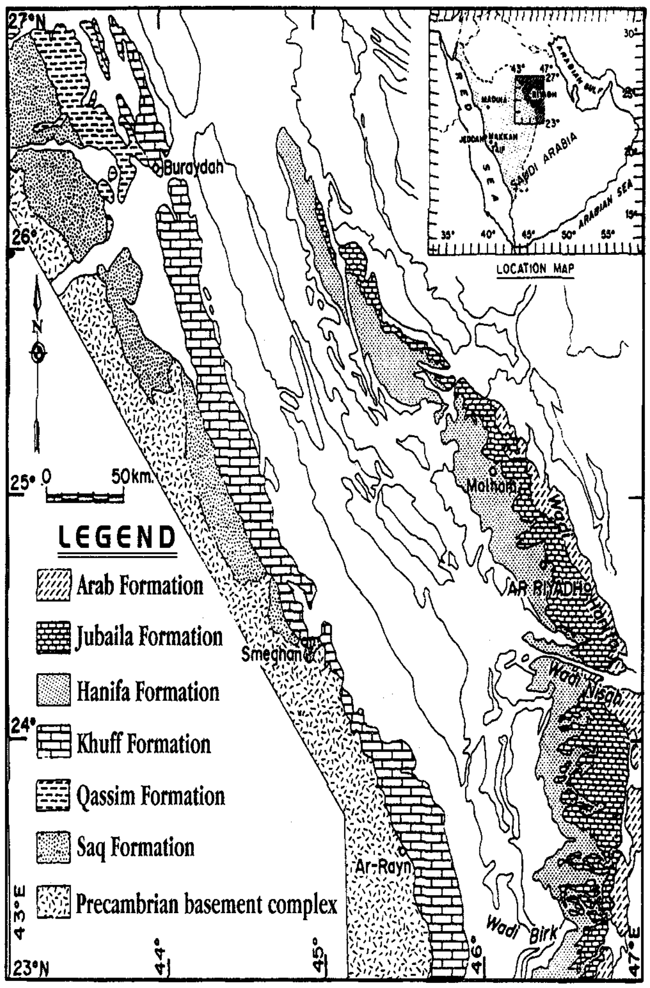


Figure (1) Geological map showing the location of the studied localities of Khuff and Jubaila Formatios (modified after Basyoni et al., 1992).

MATERIAL AND METHODS

Based on X-ray diffraction analysis , Basyoni et al. [11] showed that calcite and dolomite are the only present carbonate minerals in the Khuff and Jubaila formations. On the basis of their results, eighteen representative carbonate samples have been selected for the present thermal analysis study. The samples represent six localities (Khuff Formation: 4 from Ar Rayan, 4 from Smeghan and 2 from Buraydah; Jubaila Formation : 6 from Wadi Malham and one from each of Wadi Hanifa and Wadi Birk, Fig. 1 .

These samples have been reanalysed by X-ray diffraction to estimate the percentages of calcite and dolomite and by chemical analysis to estimate the total carbonate and insoluble residue percents . Their same powders are also used for the thermal derivatographic analysis. The use of the same sample powders in the different analyses helps to make accurate comparison and testing the effectiveness and reliability of the results of the quantitative estimation of calcite and dolomite by thermogravimetric analysis.

The studied samples are petrographically identified using Folk's classification [12 & 13] in which the state of crystallization of carbonate minerals (either micrite or sparite) and type of allochems are involved in terminology . This facilitates the investigation of the effect of the type and nature of orthochems and allochems on the thermal behaviour of carbonate minerals (e.g. sharpness and temperature of peaks).

A Hungarian- made derivatograph manufactured by MOM, Budapest was used in conducting the DTA, DTG and TG analyses . In all the studied samples , one gram of the - 200 mesh powdered sample was heated from room temperature to 1000°C in an atmosphere of air with a heating rate of 10°C per minute using ignited alumina as inert material . DTA & DTG sensitivities are 1/20 & 1/10 respectively and TG scale is 500 mg.

Results and Discussion

The results of derivatographic analysis of the studied carbonate samples are given in table (1) and their derivatograms are illustrated in figs. 2 to 6. The thermal effects shown in these results indicate the presence of three types of rocks varying in their mineralogical composition. These types are:

a- Limestone , composed of calcite.

b- Dolomite rock, formed of dolomite.

c- Dolomitic limestone to calcareous dolomite , made up of calcite and dolomite in different proportions.

All these types of rocks contain rare to few amounts of non-carbonate minerals like quartz and iron oxide minerals as indicated from chemical and petrographic analyses .

It is well known that endothermic decomposition with the liberation of carbon dioxide gas is the characteristic property of carbonates. Such decomposition as shown by DTA , DTG and TG curves can be interpreted for the three rock types as follows :

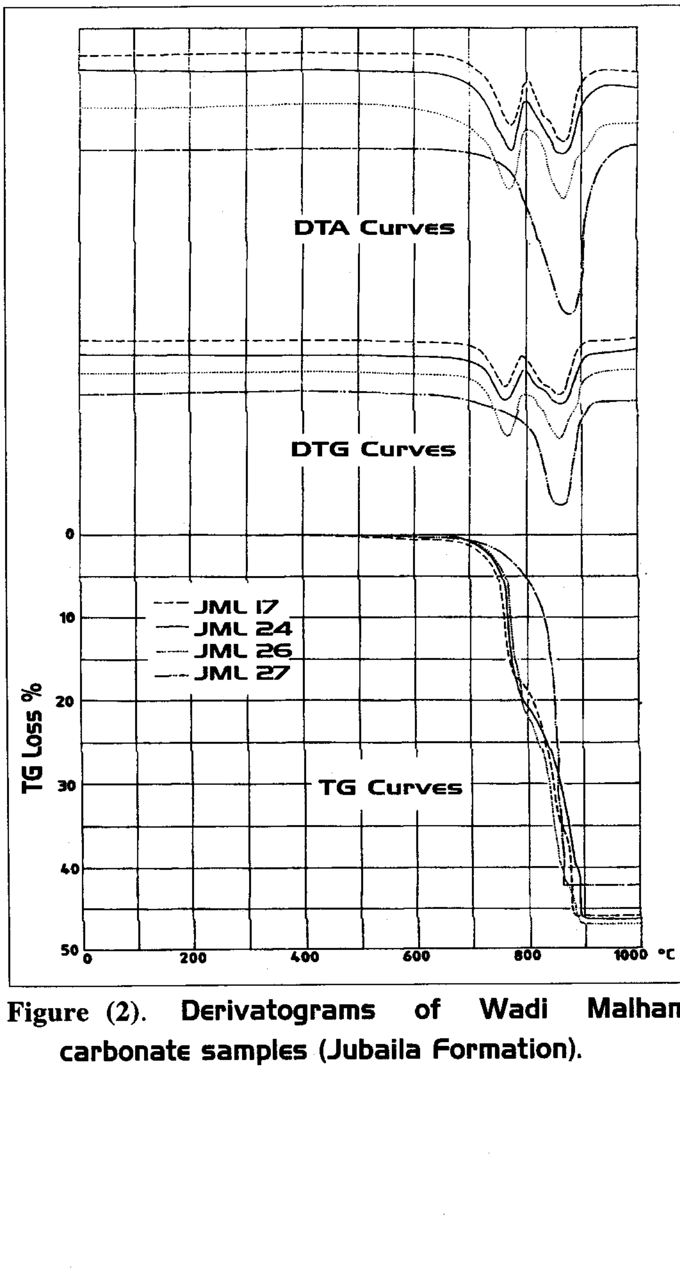


Figure (2). **Derivatograms of Wadi Malham carbonate samples (Jubaila Formation).**

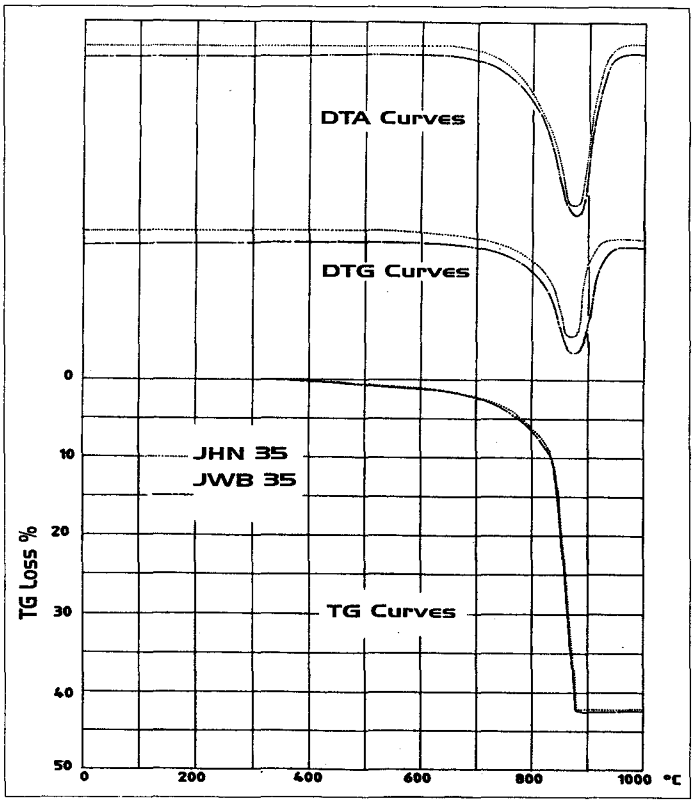


Figure (3). Derivatograms of Wadi Hanifa and Birk carbonate samples (Jubaila Formation).

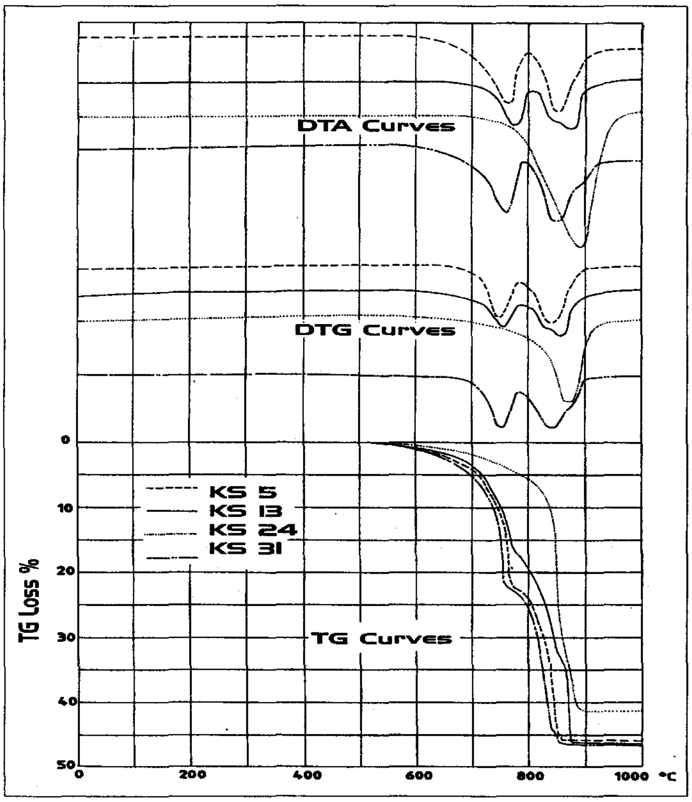


Figure (5). Derivatograms of Smegham carbonate samples (Khuff Formation).

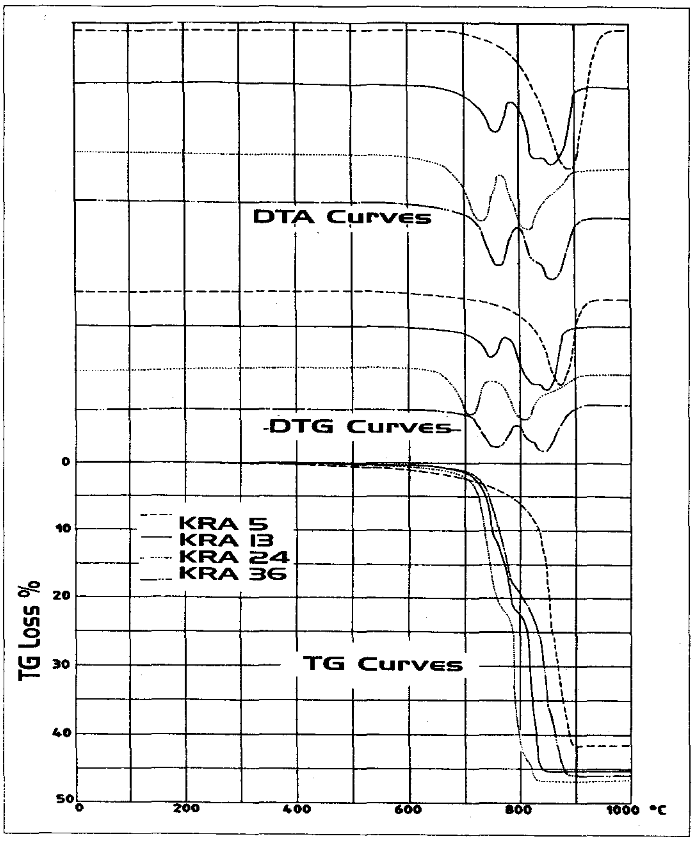


Figure. (4). Derivatograms of Ar - Rayan carbonate samples (Khuff Formation).

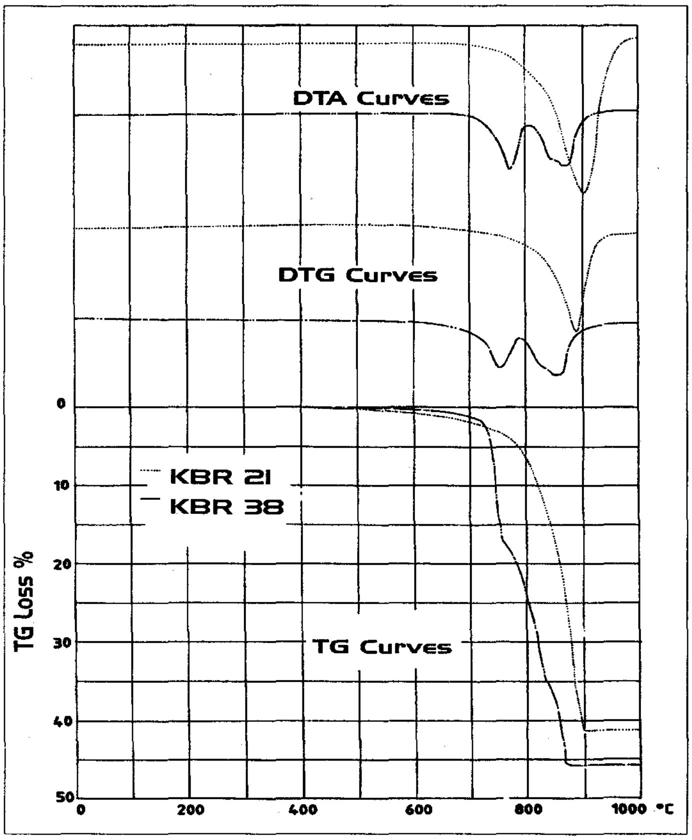
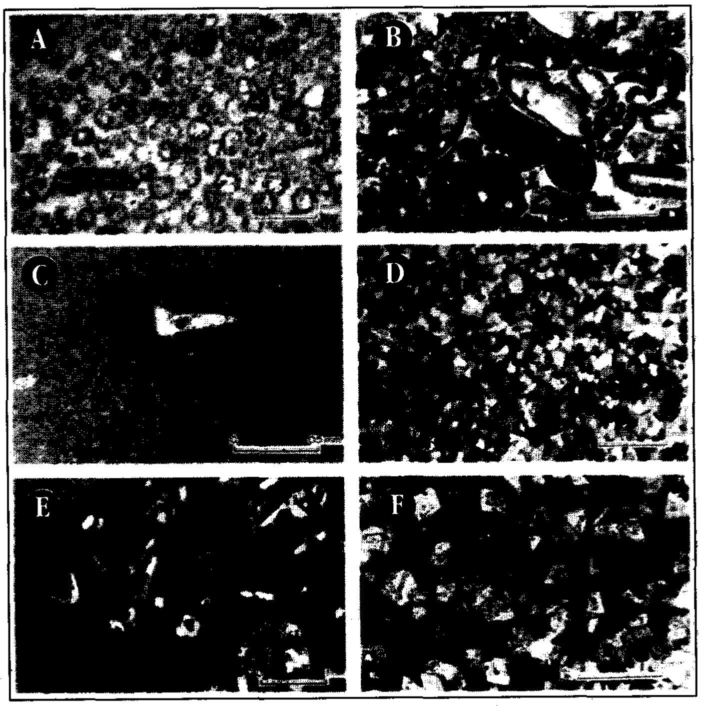


Figure (6). Derivatograms of Buraydah carbonate samples (Khuff Formation).

Samples of this type show a large, symmetric , sharp endothermic DTA peak occurring at 875° to 900° C , accompanied by a similar but smaller endothermic DTG peak at 865°C to 885°C and TG loss percent ranging form 41.65 to 42.84% of the original sample weight (Table 1 and Figs 2 to 6) . This reaction represents the decomposition of calcite ($CaCO_3$)to CaO with the liberation of CO_2 gas.

The highest temperature of this reaction is recorded in sample KBR21 (bio-oosparite, Fig. 7A) in which coarse crystalline calcite forms the main part. On the other hand , the lowest temperatures are recorded in sample JML27 (intra- biomicrite , Fig. 7B) which is made up mainly of coarse unpacked fossils and intraclasts with few scattered quartz grains and by sample JHN35 (sparse biomicrite, Fig. 7C) which is made up of micrite with sparse micro sparitic fossil patches.



This indicates that calcite when present in crypto- to micro-crystalline state or associated with other minerals (impurities like quartz), its decomposition temperature will be appreciably lowered. Todor [14] showed that decomposition temperature values for simple pure calcite mineral may reach 930° to 940° C, but when it is present in a mixture in proportions of only 10 to 15 % , the maximum of the thermal effect will then be depressed at about 800°C. Derivatograms given by Liptay [15] for pure calcite and dolomite minerals also show higher decomposition termperatures (920°C &780°, 920°C for calcite and dolomite, respectively) than those recorded in the present work.

A - Oosparite (sample KBR 21, P.P.L.) . Oolites with few molluscan shell fragments are surrounded by intergranular sparry hypidio to xenotopic calcite rhombs.

B - Intra -biomicrite (sample JML 27, P.P.L.). Abundant un-packed micritic allochems (coarse bioclasts, e.g., algal filaments , foraminifers , ostracods, molluscs and intraclasts), parts of them are dissolved creating rhombohedral pores Tiny scattered quartz grains are observed .

C - Sparse biomicrite (sample JHN 35 , C.N.). Micrite with sparse microsparitic fossil patch and tiny quartz grains.

B- Dolomite

Decomposition of dolomite is represented by two endothermic reactions; the first corresponds to the decomposition of carbonate ions associated with Mg and the second to the decomposition of those associated with Ca.

The first reaction is represented by moderate , more or less symmetric endothermic DTA peak occurring at 720° to 775° C accompanied by a similar DTG peak at 710° to 763° C and TG loss percent ranging from 21.74 to 22.36 % of the original sample weight (Table 1 and Figs 2 - 6).

The second reaction is represented by a moderate to large symmetric to asymmetric endothermic DTA peak occurring at 810° to 870° C accompanied by a similar DTG peak occurring at 800° to 862° C and TG loss percent ranging from 22.10 to 22.74% of the original sample weight (Table 1 and Figs 2-6).

In fact the samples considered to belong to this rock type (dolomite) contain minor amount of free calcite which is difficulty observed from the small shoulder at the high temperature side of both the DTA and DTG peaks and its corresponding minor TG loss (e.g. sample KS5, Fig.5).

It is worthmentioning that the highest temperatures of both reactions are recorded in sample JML26 (dolosparite , Fig.7D) which is made up totally of coarse crystalline dolomite rhombs, while the lowest temperatures are recorded in sample KRA24 (bio-dolomicrite, Fig.7E) which is made up mainly of microcrystalline dolomite with some sparitic fossils . Sample JML26 also gave the sharpest peaks due to its higher degree of crystallinity. Dolomite rhombs of sample JML17 (Fig. 7F), in spite of their coarser character than those of sample JML26 (Fig. 7D), however their decomposition peaks are less sharper and occur at relatively lower temperature (Fig. 2) due to their association with appreciable amount of micritic calcite and probably due to their perfect zoning.

D - Dolosparite (sample JML 26, C.N.). Xenotopic to hypidiotopic, cloudy unzoned sparitic dolomite with common intercrystalline pores partly filled with iron oxide.

E - Bio-dolomicrite (sample KRA 24, C.N.). Microcrystalline dolomite with sparitic calcareous algae, ostracods and pelecypod fragments .

F - Calcareous dolosparite (sample JML17 , C.N.). Coarse crystalline zoned idiotopic dolomite rhombs with common micritic calcite (stained with Alizarin Red S) and rare bryozoa.

C. Dolomitic limestone - calcareous dolomite

Most of the studied samples belong to this type . They gave the same first reaction as in dolomite samples but with lesser peak areas and TG loss percents depending on the amount of dolomite mineral. Of course a proportional relation exists between these two variables and dolomite concentration.

The second reaction of the decomposition of Ca CO_3 is different here and is represented by double , asymmetric DTA peak (810° to 870°C and 850° to 900° C) accompanied by double endothermic DTG peak (800° to 862°C and 850° to 890°C) and TG loss percent ranging from 11.9 to 22.74 % and 0.79 to 23.25% of the original sample weight, respectively. The first thermal effect of this double peak corresponds to the Ca CO_3 bonded in the dolomitic lattice and the second corresponds to the unbonded Ca CO_3 (i.e. free calcite). The peak of the latter is relatively larger in samples containing appreciable amount of free calcite as KRA 13 & 36(Fig.4), KS13 (Fig.5), KBR38 (Fig. 6) and JML17 &24 (Fig. 2), while it appears as a shoulder in those containing lesser amount as KRA24 (fig. 4), KS31 (Fig.5)and JML26 (Fig. 2).

In all studied dolomite samples it is found that the ratio of CO_2 in $MgCO_3$/CO_2 in Ca CO_3 is less than unity (Table 1) indicating that dolomite is of the calcitic type . If the ratio is higher than unity, the dolomite is termed magnesian dolomite. In this case it is possible that it should contain $MgCO_3$, which is not bonded into the dolomitic lattice , and then , at about 600°C there will appear a thermal effect with a loss of mass, corresponding to the percentage of magnesite contained in the dolomite . Such thermal effect is not recorded in the studied samples . It is worthmentioning that the presence of Mg CO_3 or Ca CO_3 outside the lattice of dolomite fails to be established from the percentage of the chemical analysis, this is possible only from the thermal curves.

Quantitative Estimation of Calcite and Dolomite By Thermal Derivatography

The method used here is based on measuring the loss percent of mass due to the decomposition of $CaCO_3$ and $MgCO_3$ and evolution of their bonded CO_2 on the TG curve. Pure limestone shows smooth TG curve while in dolomitic limestone and calcareous dolomite samples it shows two deflections dividing the curve into three parts . From the low temperature part the percentage of CO_2 of Mg CO_3 is estimated while CO_2 percentage of $CaCO_3$ bonded in dolomite is estimated from the middle part and that of the free calcite is estimated from the high temperature part. DTG curve is very helpful in exact estimation of such percentages .

A- Limestone

In limestone samples (e.g. sample KRA5, Table 1 and Fig.4) the calcite percentage (z) is estimated as follows using the percentage of CO_2 released from Ca CO_3 :

Ca CO_3		Ca O + CO_2
100.09 (mol. wt.)	$\xrightarrow{>900\ C}$	44.01 (mol. wt.)
Z	————	41.66

$$\text{Calcite \% (Z)} = \frac{100.09 \times 41.66}{44.01} = 94.75\%$$

B. Dolomite

In dolomite rock samples (e.g. sample KS5, Table 1, and Fig.5) the dolomite percentage (x) is estimated as follows using the percentage of CO_2 released from $MgCO_3$:

$$Ca\,Mg\,(CO_3)_2 \xrightarrow{\sim 775°\,C} CaCO_3 + MgO + CO_2$$

184.42 (mol. wt.)	————	44.01
X		22.36

Dolomite % (X) = $\frac{184.42 \times 22.36}{44.01}$ = 93.69 %

C- Dolomitic limestone - Calcareous dolomite

In samples containing appreciable amount of dolomite and calcite (e.g. KRA13,Table 1 and Fig.4) both minerals are estimated using the following steps:

1- Determination of dolomite (X):

$$Ca\,Mg\,(CO_3)_2 \longrightarrow Ca\,CO_3 + MgO + CO_2$$

184.42	————	44.01
X	————	10.80

Dolomite % (X) = $\frac{184.42 \times 10.80}{44.01}$ = 45.26 %

2- Determination of calcite resulting from dolomite (Y):

The estimated percentage of dolomite is used here.

$$CaMg\,(CO_3)_2 \longrightarrow CaCO_3 + MgO + CO_2$$

184.42	————	100.09
45.26	————	Y

Calcite (from dolomite)%(Y) = $\frac{100.09 \times 45.26}{184.42}$ = 24.62%

3- Determination of total calcite (Z):

The TG loss % corresponding to the release of CO_2 from Ca CO_3 in the dolomite lattice and free calcite is used here

$$CaCO_3 \longrightarrow CaO + CO_2$$

100.09	————	44.01
z	————	34.44

Total calcite % (z) = $\frac{100.09 \times 34.44}{44.01}$ = 78.33 %

Free calcite % = 78.33 - 24.62 = 53.71%

Similar result is directly obtained for free calcite when estimated using the last part of TG loss % corresponding to the release of its CO_2 content as follows :

100.09	————	44.01
F.C.	————	23.25

Free calcite % = $\frac{100.09 \times 23.25}{44.01}$ = 52.87 %

This last method can be applied for samples containing appreciable amount of free calcite because the TG loss% corresponding to the release of its CO_2 can be read easily on the TG curve . The other method is preferably used for samples containing lesser amount of free calcite .

To sum up the results of all these calculations, together with those obtained from XRD and chemical analyses are given in table 2 . The close similarity between these results prove the effectiveness and reliability of thermogravimetric analysis for quantitative estimation of calcite and dolomite.

Table 1. Thermal analysis results of the different types of the Jubaila & Kuff carbonate rocks.

a) Jubaila Formation

Locality	Sample No*	Endothermic Peak		TG Loss %	Description of DTA peak	Assignment of the reaction	Rock name and % of sparite
		DTA°C	DTG°C				
Wadi Malham	JML 17	765 830 870	757 825 860	17.66 17.95 10.73	moderate, symmetric large, double, asymmetric	decomposition of Mg Co3 dec. of Ca Co3 in dolomite dec. of free Ca Co3	Sparsely fossiliferous dolosparite (zoned) dolosparite 75% micrite 25%
	JML 24	767 828 870	758 820 860	19.76 20.12 6.58	moderate, symmetric large, double, asymmetric	dec. of Mg Co3 dec. of Ca Co3 in dol. dec. of free Ca Co3	Sparsely fossiliferous dolosparite dolosparite 80%
	JML 26	775 870 900	763 862 890	21.89 22.26 2.85	mod.- large, sym., sharp large, double, asym.	dec. of Mg Co3 dec. of Ca Co3 in dol. dec. of free Ca Co3	Dolosparite dolosparite 90%
	JML 27	875	865	42.23	large, sym., sharp	decomposition of Ca Co3	Intra-biomicrite with un packed allochems and tiny quartz grains.
	JML32**	768 830 872	760 820 862	21.77 22.14 1.49	mod.- large, sym., sharp large, double, asym.	dec. of Mg Co3 dec. of Ca Co3 in dol. dec. of free Ca Co3	Dolosparite dolosparite > 80%
	JML35**	760 822 865	750 815 855	21.82 22.19 0.79	mod.- large, sym., sharp large, double, asym.	dec. of Mg Co3 dec. of Ca Co3 in dol. dec. of free Ca Co3	Dolomicrosparite with tiny quartz grains
Wadi Hanifa	JHN 35	875	865	42.70	large, sym., sharp	dec. of Ca Co3	Sparse biomicrite no sparite.
Wadi Birk	JWB 35	880	870	42.84	large, sym., sharp	dec. of Ca Co3	Biomicrite no sparite

* Sample numbers used here are the same as those given in Basyoni et. al. [11] .

** Derivatograms of these two samples are very similar to that of sample JML 26.

Table 1 (Continued)

b) Kuff Formation

Locality	Sample No*	Endothermic Peak DTA°C	DTG°C	TG Loss %	Description of DTA peak	Assignment of the reaction	Rock name and % of sparite
Ar-Rayan	KRA5	880	870	41.66	large, sym., sharp	dec. of Ca Co3	Micrite (after dolomite, i. e., dedolomite)
	KRA13	750 830 860	742 822 850	10.80 11.19 23.25	mod, sym, sharp large, double, asymmetric	dec. of Mg Co3 dec. of Ca Co3 in dol. dec. of free Ca Co3	Dolomicrosparite sparite 20%
	KRA24	720 810 855	710 800 850	21.74 22.10 3.29	mod.- sym, sharp large, double, asym.	dec. of Mg Co3 dec. of Ca Co3 in dol. dec. of free Ca Co3	Bio- dolomicrite sparite 10%
	KRA36	755 820 850	750 812 840	17.66 17.95 10.55	mod.-, sym., large, double, asym.	dec. of Mg Co3 dec. of Ca Co3 in dol. dec. of free Ca Co3	Bio- dolomicrite sparite 10-15%
Smeg-han	KS5	760 847 880	750 840 870	22.36 22.74 0.9	mod.- sym, sharp large, sym., sharp minor shoulder	dec. of Mg Co3 dec. of Ca Co3 in dol. dec. of free Ca Co3	Dolomicrite no sparite
	KS13	770 840 870	760 830 860	16.48 16.76 13.16	mod.- large, sym., sharp large, double, asym.	dec. of Mg Co3 dec. of Ca Co3 in dol. dec. of free Ca Co 3	Dolomitic biomicrite-sparite transition
	KS24	890	875	41.65	large, sym., sharp	dec. of Ca Co3	Biomicrite- sparite 30%
	KS31	760 845 880	750 838 870	21.89 22.26 2.72	mod.- large., sym. large, double, asym.	dec. of Ca Co3 in dol. dec. of free Ca Co3 dec. of Ca Co3	Dolomicrite- micro-sparite no sparite
Bura-ydah	KBR21	900	885	41.67	large, sym., very sharp	dec. of Ca Co3	Bio- oosparite
	KBR38	770 840 870	760 830 860	17.13 17.43 11.40	mod., sym., sharp large, double, asym	dec. of ng Co3 dec. of Ca Co3 in dol dec. of free Ca Co3	Dol-oosparite sparite 85%

Table 2. Results of chemical, X-ray diffraction and thermogravimetric analyses of the Jubaila and Khuff carbonate rocks.

			Chemical analysis data		XRD analysis data		Thermogravimetric analysis data			
Formation	Locality	Sample No.	Total cabonate %	I .R. %	Calcite %	Dolomite %	Total calcite %	Calcite from dolomite %	Free calcite %	Dolomite %
JUBALLA	Wadi Malham	JML17	97.41	2.30	24.41	74.82	65.23	40.16	25.07	74.00
		JML24	99.26	1.03	14.96	83.79	60.72	44.94	15.78	82.80
		JML26	97.48	2.99	6.48	92.76	57.11	49.78	7.33	91.73
		JML27	98.83	1.19	96.66	2.85	95.87	--	95.87	--
		JML32	94.91	2.02	3.39	92.25	53.74	49.51	4.23	91.23
		JML34	96.39	2.21	1.79	92.46	52.26	49.62	2.64	91.43
	Wadi Hanifa	JHN35	97.11	2.20	97.16	0.00	97.11	--	97.11	--
	Wadi Birk	JWB35	97.50	2.30	97.43	0.00	97.43	--	97.43	--
KHUFF	Ar-Rayan	KRA5	96.29	2.90	94.76	2.33	94.75	--	94.74	--
		KRA13	98.00	2.60	52.88	46.20	78.33	24.62	53.71	45.26
		KRA24	99.72	0.44	7.48	92.11	57.70	49.44	8.26	91.10
		KRA36	98.01	0.36	23.99	74.81	64.82	40.16	24.66	74.00
	Smeghan	KSS	97.24	1.53	2.08	94.76	53.76	50.85	2.91	93.69
		KS13	98.40	0.41	29.92	69.82	68.05	37.48	30.57	69.06
		KS24	97.98	0.52	94.76	2.32	94.75	--	94.75	--
		KS31	97.58	1.63	6.18	92.76	56.81	49.78	7.03	91.73
	Buraydah	KBR21	97.87	1.41	94.76	3.01	94.75	--	94.75	--
		KBR38	99.68	0.64	25.93	72.61	65.57	38.96	26.61	71.28

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