DISSOLUTION OF " FER DE LANCE" GYPSUM VARIETY KINETIC APPROACH

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ABSTRACT

9 grams of 90-240 μm grain-size particles of gypsum in "fer de lance" were suspended in 500 ml of reagent solution (water, $NaHCO_3$ 10^{-3} N), stirred in a flask at 25 °C. The suspension was sampled through the time to insure the progress of dissolution. The dissolution curves of SO_4^{2-} and Ca^{2+}, determined by chromatography ionic, showed a difference in the magnitude of the release of these two components of gypsum, and indicated that bicarbonate most likely enhances the dissolution. The analyses of experimental data using Davies and Transition State Theories indicated that the kinetic dissolution of gypsum especially in water seems to be a second order dependent process of surface reaction. The Bovengton-Jones plot is correctly verified for the experimental points succeeding the initial step of dissolution. The kinetic parameters determined by the application of Transition Theory may be extended to interpret the non adjustment of Bovengton-Jones plot in the initial stage of dissolution as an apparent fact due to the non linearity between the net rate of reaction v_{ne} and the reaction free energy ΔG.

Keywords: Gypsum, dissolution, kinetic, activation energy, transition state theory, diffusion, chemical reaction, reaction rate.

INTRODUCTION

Gypsum may be a major component of soil especially in arid regions. It may expand to affect soils of ustic and xeric regimes (Nettleton *et al*;1982). The unsuitable properties, physical and chemical, induced by its dominance have been incited, here and there, an extensive efforts to promote a general policy dealing with the big problems inferred by this component. Due to its behavior as a product of an intermediate solubility, the dynamic of gypsum in soil seems to be a complex process difficult to be controlled. The dynamic character of gypsum dissolution in gypsiferous soils has been recently shown by Agib and Bourrié (2000). The thermodynamic calculations indicate that one reason of these dynamic character is the paragenetic interrelationship between gypsum and calcite which seems to be the main process responsible for gypsum equilibrium. But the intrinsic factor giving this dynamic character to gypsum transformation is directly related to the kinetic processes of gypsum dissolution and precipitation. The occurrence of these processes in soil between the successive irrigation practices stand to be the most effective factor responsible of gypsum distribution within and among soil profile. The impact of gypsum dissolution and precipitation is extended to affect karst phenomenon and underground voids and cavities evolution. Due to the importance of these phenomenon, especially as a potential risk of subsidence, karst kinetic has been a subject of modeling by a number of studies Dreybrodt (1996). Some kinetic aspects of gypsum dissolution and precipitation have been studied by different authors. Even, the processes of dissolution and precipitation of gypsum, constitute two phenomenon completely reversible Zooggari (1996). Each process might have its own

kinetic characters Bonneau (1981) depending on crystalline properties Barton and Wild (1971) and particles sizes Agib and Bourrié (1996). In this communication, the kinetic of dissolution of a variety of gypsum " fer de lance" in two medium, water and sodium bicarbonate solution, has been approached using the diffusion, reaction, and transition state theories.

THEORITICL CONSIDERATIONS

The dissolution may be considered as
- an irreversible process controlled by the diffusion of solute between the surface and the bulk solution ;
- an irreversible process controlled by the surface reaction ;
- a reversible process of direct and reverse half reactions with a transition activated complexes .

a- **Control by diffusion:** The diffusion theory, first developed by Nernst, is based on Fick first law :

$$\frac{dC}{dt} = K_D(C_o - C) \tag{1}$$

where C_o is the solute concentration at the surface reaction , for a mineral in dissolution this concentration is arbitrary taken as equivalent to saturation one, C is the solute concentration in the bulk solution, and K_D is the diffusion constant defined by the expression

$$K_D = \frac{DS}{V\delta} \tag{2}$$

in this equation D is the diffusion coefficient, S is the surface, V is solution volume, and δ represent the thickness of an immobilized layer disjoining surface from solution Delmas (1979). The integration of equation (1) gives :

$$K_D = \frac{1}{t} Log\left(\frac{C_o}{C_o - C}\right) \tag{3}$$

b- **Control by surface reaction :** This theory of Davies and Jones (1955) considers the dissolution as a process controlled by the rate of surface reaction expressed by the following equation :

$$\frac{dC}{dt} = K_s(C_o - C)^n \tag{4}$$

K_s is the rate constant of surface reaction, and n is the order of reaction, which may be : 1,2,3 and rarely more than that. The general integral form of the last equation is:

$$K_s = \frac{1}{n-1}\left[\frac{1}{(C_o - C)^{n-1}} - \frac{1}{C_o^{n-1}}\right]\frac{1}{t} \tag{5}$$

If dissolution is controlled by diffusion, K_D will be independent of time, then the graphical representation K_D, t) of equation (3) shows a straight line parallel to t axes. But when it is controlled by the surface reaction, K_s will be the independent parameter, and the graphical representation (K_s ,t) of equation (5) shows a straight line parallel to t axes.

c- Dissolution as forward and reverse process – the transition state model: This model is derived from the transition state theory. For an overall reaction:

$$A + B \xrightarrow[\;2\;]{\;1\;} C + D \tag{6}$$
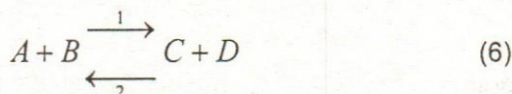
it is possible to write:

$$v_1 = k_1 C_A C_B \tag{7} \qquad , \qquad v_2 = k_2 C_C C_D \tag{8}$$

v_1 is the rate of forward reaction, and k_1 is its rate constant, v_2 is the rate of the reverse reaction, and k_2 is its rate constant. The tow rates constants are defined by the Transition State Theory by the tow expressions:

$$k_1 = \frac{kT}{h} \frac{\gamma_A \gamma_B}{\gamma^+} e^{-(\Delta G^{+1}/kT)} \tag{9} \qquad k_2 = \frac{kT}{h} \frac{\gamma_C \gamma_D}{\gamma^+} e^{-(\Delta G^{+2}/kT)} \tag{10}$$

where: h Planck's constant, k Boltzmann's constant, T temperature in Kelven, γ activity coefficient, γ^+ the activity coefficient of an activated complex, ΔG^{+1} is the standard free energy difference between the activated complex and the reactants A,B:

$$\Delta G^{+1} = G_1^{o+} - G_A^o - G_B^o \tag{11}$$

Likewise, $$\Delta G^{+2} = G_2^{o+} - G_C^o - G_D^o \tag{12}$$

The ratio of the rate constants can be obtained from (9) and (10):

$$\frac{k_1}{k_2} = \frac{\gamma_A \gamma_B}{\gamma_C \gamma_D} e^{-(\Delta G^{+1} - \Delta G^{+2})/kT} = \frac{\gamma_A \gamma_B}{\gamma_C \gamma_D} e^{-\frac{\Delta G^o}{kT}} e^{\frac{G_2^{o+} - G_1^{o+}}{kT}} \tag{13}$$

$$-\Delta G^o = G_A^o + G_B^o - G_C^o - G_D^o$$

If the activated complex is the same in either direction equation (13) may be simplified

$$\frac{k_1}{k_2} = \frac{\gamma_A \gamma_B}{\gamma_C \gamma_D} e^{-\frac{\Delta G^o}{kT}} \tag{14}$$

From equations (7),(8) and (13)

$$\frac{v_1}{v_2} = \frac{k_1 C_A C_B}{k_2 C_C C_D} = \frac{\gamma_A \gamma_B}{\gamma_C \gamma_D} \frac{C_A C_B}{C_C C_D} e^{-\frac{\Delta G^o}{kT}} = \frac{a_A a_B}{a_C a_D} e^{-\frac{\Delta G^o}{kT}} e^{\frac{\Delta G^{o+}}{kT}} = e^{-\frac{\Delta G}{kT}} \tag{15}$$

$$\Delta G^{o+} = G_2^{o+} - G_1^{o+}$$

where ΔG is the actual free energy difference. Equation (15) can be put also in the useful form

$$v_{net} = v_1 \left(1 - e^{\frac{\Delta G}{RT}}\right) \tag{16}$$

where v_{net} is the net rate reaction, and is the difference between the forward and reverse rates, $v_{net} = v_1 - v_2$. The equivalent form of equation (14) for gypsum dissolution is

$$\frac{v_1}{v_2} = \frac{a_{CaSO_4.2H2O}}{a_{Ca^{2+}} \cdot a_{SO_4^{2-}}} e^{-\frac{\Delta G^o}{RT}} e^{\frac{(G_2^{0+} - G_1^{0+})}{RT}} = e^{-\frac{\Delta G}{RT}} \qquad (17)$$

Therefore, the net rate of gypsum dissolution can be correlated to the forward reaction rate by the following equation

$$v_{net} = v_1 (1 - \frac{a_{Ca^{2+}} a_{SO_4^{2-}}}{a_{CaSO4..2H2O}} e^{\frac{\Delta G^o}{RT}} e^{\frac{-\Delta G^{o+}}{RT}}) = v_1 (1 - e^{\frac{\Delta G}{RT}}) \qquad (18)$$

$$a_{CaSO4.2H2O} = 1$$

MATERIALS AND METHODS

A sample of gypsum in "fer de lance" is reduced to a powder, and separated to different grain-size classes. The sheet grain-size 90-240μm was retained to study the dissolution process. 9 g of powdered gypsum was weighed and suspended in 500 ml of the reagent solution (water, NaHCO$_3$ 10^{-3} N), stirred in a flask integrated to a pH-meter and conductimetric system . The suspension ratio is pre-chosen to minimize the effect of S/V (S = total gypsum surface, V = volume of suspension) change. 5 ml of suspension have been sampled each time and filtered using a Millipore^{TM} , the analysis of filtrates was performed by ionic chromatography.

RESULTS AND DISCUSSION

The curves representing Ca^{2+} and SO$_4$ release among the time (fig.1) can be represented by an empirical logarithmic function of time

$$C_{(t)} = a \ln t - b$$

where a, b are constants, and $C_{(t)}$ is the concentration of Ca^{2+} or SO$_4^{2-}$ measured at a given time t. It seams that this equation fit best dissolution in water($R^2 \approx 0.85$) than dissolution in bicarbonate medium ($R^2 \approx 0.98$).
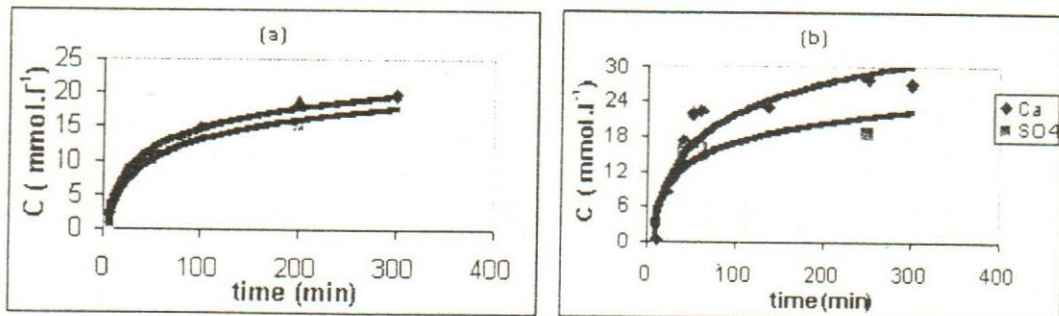

Fig.1. Evolution of dissolved calcium and sulfate concentrations as a function of time t : in water (a) and in NaHCO$_3$ 10^{-3}MI^{-1} (b).

Although the release of calcium and sulfate has the same general trend, nevertheless the two species should be distinguished each one of the other by the magnitude of dissolution. Calcium appears to be more dissolved than sulfate especially in bicarbonate medium, where the dissolution appears to be gently enhanced by this electrolyte. A simple calculation of ionic product $(Ca^{2+})(SO_4^{2-})$ indicates, in comparison with gypsum solubility product, that the dissolution process maintained an over saturation state. From a thermodynamic viewpoint, this case of over saturation represents a transient state, and the equilibrium is attended to be established at a given time. The advancement of dissolution in bicarbonate medium show the passage of process from an increasingly gypsum dissolution phase to a second phase marked by gentle decrease of ionic product due essentially to a substantial decrease of calcium concentration.

K_T AND K_S AS A PARAMETER OF KINETIC

Kinetic as a function of Ca^{2+}, SO_4^{2-} - Equation (3) was used first to test the Nernst theory and to verify if the kinetic is under the control of diffusion process or not. The results given in fig. illustrate how K_T does change with time , and permit to conclude that diffusion appears not to be the controlling factor of dissolution. Assuming gypsum dissolution as a reaction of second order, the kinetic should then obey equation(5). The adjustment of this equation without any presumption about C_o may be represented by the Bovington and Jones function (1970)

$$\frac{C}{t} = -SK_S C_o C + SK_S C_o^2 \qquad (19)$$

In this equation C becomes a linear function of C/t , so a plot of C versus C/t permit easily to verify if the reaction is of a second order and to adjust in the same time the reaction constant SK_S. Furhermore, the extrapolation of this plot to intercept the abscissa axes for C/t = 0 gives C_o which may be different of thermodynamic solubility, the values depicted by this extrapolation are 18.22 and 18.4 m.mol l^{-1} for SO_4^{2-} in water and in bicarbonate medium, the equivalent concentration for Ca^{2+} are 25 and 31.3 m.mol l^{-1}. It must be noted that the initiation of process is excluded from this adjustment because this stage of dissolution may be altered by a parasitic effect due to gypsum sample grinding. Fig.2. illustrates the results conducted by this adjustment.

It can be seen the well adjustment of Bovengton-Jones function especially in water medium. This adjustment indicates that the Davies-Jones theory is reasonably verified that it should be likely to consider gypsum dissolution as a reaction of second order especially for dissolution in water, and to treat its kinetic as a dependent function of reaction. The reaction seams to have the same order whatever the component of gypsum, calcium or sulfate, undertaken for the adjustment.
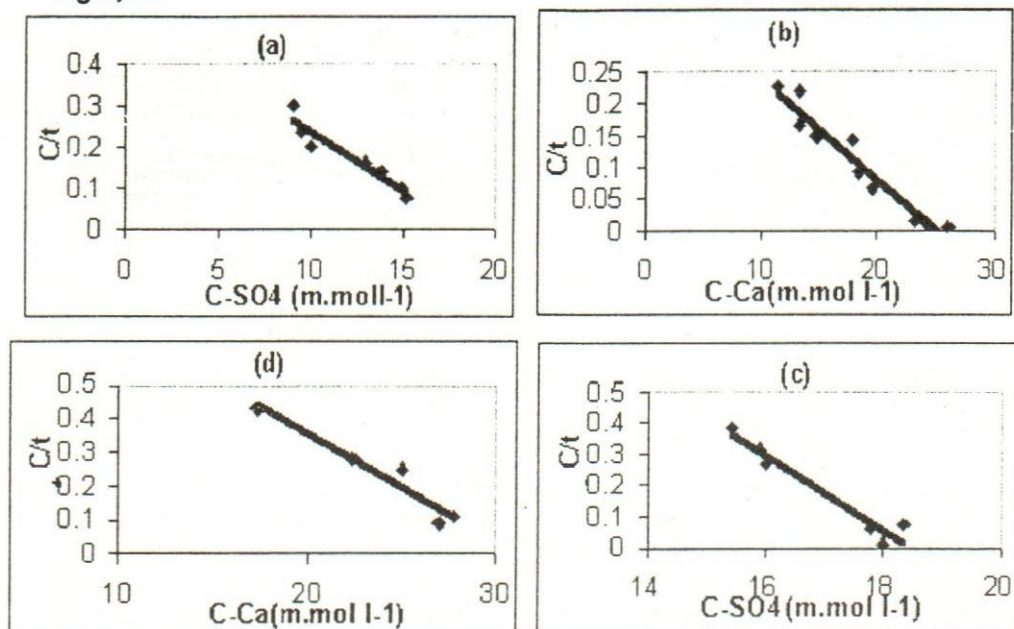
Fig.2.Verification of Davies and Jones function for the surface reac-
tion ,a graphical representation of Bovengton and Jones equation
for; sulfate in water (a), calcium in water (b), sulfate in $NaHCO_3$ 10^{-3}
Ml^{-1} (c), and calcium in $NaHCO_3$ 10^{-3} Ml^{-1} (d).

Table (2): Kinetic coefficient SK_s calculated using the Bo-vengton-
Jones plot.

property		SO_4^{2-}	Ca^{2+}	$\sqrt{IP}$
SK_s	water	0.00158	0.00064	0.00245
$mmol.l.min^{-1}$	$NaHCO_3$	0.0064	0.00099	0.00173

Nevertheless, referring to the adjusted parameter SKs (tab.2), it
could be concluded that the tow components appear to have tow different
rates, the release of sulfate is being faster especially in bicarbonate medium.
The extrapolated concentrations determined by the Bovengton-Jones plot
was then used to estimate SK_s trying to see how this coefficient does
interrelate with time. Fig. 3. shows the complete independence of this
coefficient of time for SO_4^{2-} in water prooving the conclusion considered
above about the nature of the factor controlling the kinetic, the case of Ca^{2+}
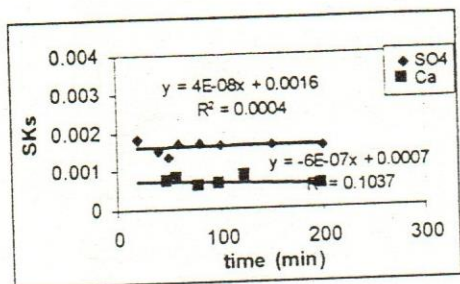was reasonably verified. For the dissolution in bicarbonate the results
seemed less evident.

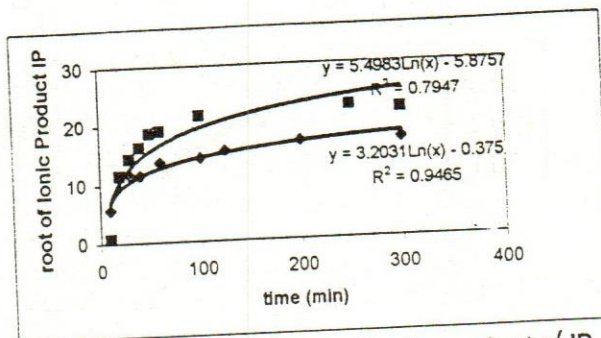Fig. 3. A plot of SK$_s$ vs time for water midium.

Fig.4.Evolution of the root of ionic product $\sqrt{IP}$ as a function of time t for the dissolution in : water ♦, NaHCO$_3$ 10^{-3} Ml^{-1} ■ .

Kinetic as a function of ionic product IP $(Ca^{2+})(SO_4^{2-})$ – The process is represented here by the variation of the root of ionic product $\sqrt{IP}$ in function of time t (fig.4).

The kinetic seems to be a logarithmic function of time, $C_{(t)} = a \ln (x) - b$, especially for the dissolution in water. The plot C/t vs C verify well the Bovengton-Jones function (fig.5). The apparent reaction constants SKs, indicate that the dissolution proceed a little bit faster in water medium.
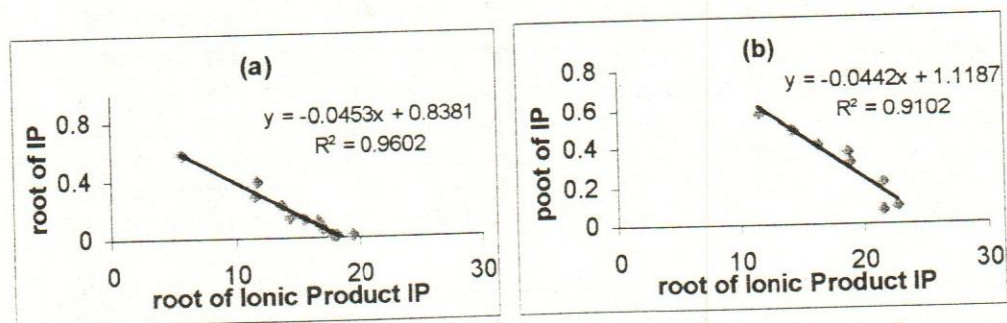

Fig.5.Verification of Davies and Jones function for the surface reaction ,a graphical representation of Bovengton and Jones equation for the dissolution in: water (a), NaHCO$_3$ 10^{-3} Ml^{-1} (b).

Free energy change :

Equation (18) has been applied to calculate the free energy change of reaction by introducing in this equation the ionic product $(Ca^{2+})(SO_4^{2-})$. The calculation was first performed considering that the activated complex is the same in either direction. This assumption means that $\Delta G^{o+} = 0$, and the second exponential term $e^{\Delta G^{o+}/RT}$ of equation becomes equivalent to unity. A simple calculation shows here that the free energy change ΔG of the reaction becomes positive very soon. This undergoes when the solution becomes saturated in gypsum and the system attains the thermodynamique equilibrium. At this stage of processes, the dissolution must be prevented according to the estimated ΔG, and it is attended that the reverse reaction becomes the dominant process. Perhaps, the experimental data show that the dissolution stills proceeding despite the over saturation state. Then, if the transition state model is applicable in this type of processes it should be reasonable to think that the previous assumption concerning the activated complex is somewhat arbitral, and it is more likely to take in consideration tow different activated complexes for the tow pathway of reaction, the direct and the reverse half reaction. Hence, the standard free energy change between the tow activated complex ΔG^{o+} may be different of zero, here it must be positive to promote the dissolution in these conditions of over saturation. To draw out the curve of the free energy change of reaction ΔG as a function of concentration C it was necessary to define ΔG^{o+}. The determination of this difference has been guaranteed assuming that the dissolution continues until the root of ionic product $\sqrt{IP}$ reaches its maximum determined by the Bovengton-Jones plot above, namely 19.3 and 25.56 m.mol l^{-1} for the dissolution in water and bicarbonate mediums respectively. At this point the net rate v_{net} approach zero and the equality $v_1 = v_2$ can be assumed. Solving equation (17) for these conditions gives 1.774 and 1.915 kcal.mol^{-1} as a values of ΔG^{o+} for the dissolution in water and in bicarbonate mediums successively. The calculation of ΔG is then accomplished by introducing these values in equation (18) and solving it for the different ionic product. As could be expected, the free energy difference ΔG (fig.6) may be represented as a logarithmic function of concentration C, $\Delta G = a \ln ((Ca^{2+})(SO_4^{2-})) - b$.

Reaction Rates- The net rate of reaction v_{net}, equivalent to dC/dt, has been calculated using the experimental data, and introduced then in equation (18) to estimate the forward rate v_1 so, by difference the reverse rate v_2 becomes defined. A plot of v_{net} and v_1 vs ΔG (fig.6) shows a linear interrelationship. This linearity may be explained supposing that the system approaches its maximum of dissolution. At this stage of processes $| \Delta G | < RT$, hence the exponential term $e^{\Delta G/RT}$ in equation (18) may be written as $(1 + \Delta G/RT)$ that this equation is expended to obtain the expression
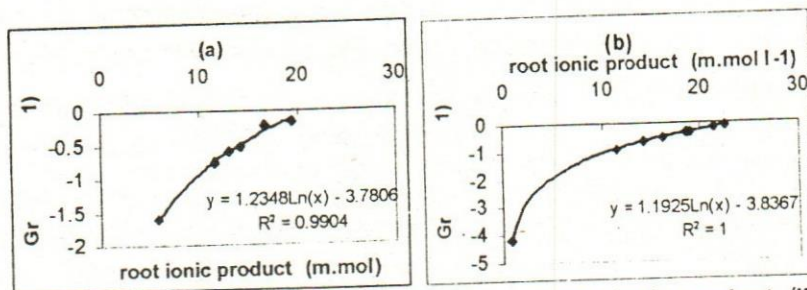
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Fig.6. Free energy change ΔG_r in function of root ionic product $\sqrt{IP}$ For the dissolution in: water (a), $NaHCO_3$ 10^{-3} Ml^{-1} (b). ΔG_r is calculated using the thermodynamic data: $\Delta G^\circ_{f\,(CaSO4.2H2O)}$ = - 430.17 , $\Delta G^\circ_{f(Ca2+)}$= - 132.52, $\Delta G^\circ_{f(SO42-)}$ =- 177.95, $\Delta G^\circ_{f(H2Ol)}$=- 56.687 kcal.mol^{-1} (Lindsay,1979).
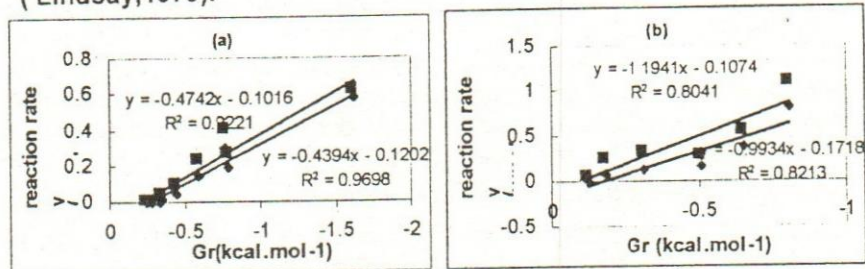

Fig.7. A plot of net rate v_{net}♦ , and forward rate v_+ ■ versus free energy ΔG_r for the dissolution in: water (a), $NaHCO_3$ 10^{-3} Ml^{-1} (b).

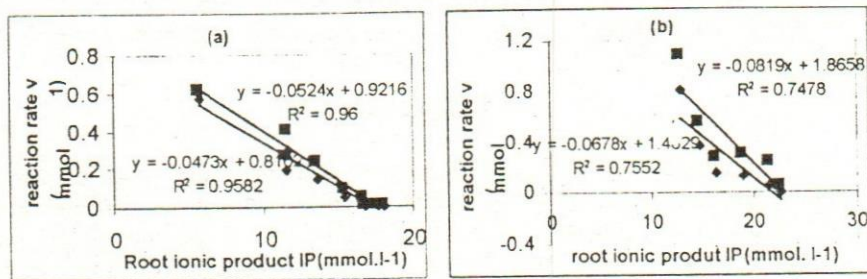Fig.8. A plot of net rate v_{net}♦ , and forward rate v_+ ■ versus root ionic product $\sqrt{IP}$ for the dissolution in: water (a), $NaHCO_3$ 10^{-3} Ml^{-1} (b).

$$v_{net} = -\frac{v_+ \Delta G}{RT} \tag{20}$$

Although this equation establishes a linear interrelationship between v_{net} and ΔG it might induce a confusion since it implicates the constancy of v_1 whereas this rate behaves similarly as a function of ΔG. This behavior is rather expected since ΔG could be bind as a driving force of reaction allowancing or not the dissolution to be more exhaustive. In this sense the free energy change ΔG is sometimes called the "affinity A" (Prigogine,1967; cited by Lasaga,1981). Equation (7) establishes also a linear interrelationship between v_1 and C, the plot v_1 vs C permits to verify this function and to obtain the rate constant k_1.   Fig.7 shows that this function is correctly verified, the two rate constants, for the dissolution in water and bicarbonate mediums, conducted by this plotting are 0.052 and 0.0819 mmol[-1].l.min[-] respectively.   A similar interrelationship between v_{net} and C might be expected also, the data given in fig.8 verify the linearity between these tow variants.

The above tow functions may be compared with Bovengton-Jones function, all of its is interpreted in the same manner and represents a criterion to determine the reaction order and to say if the kinetic of dissolution is controlled by the chemical processes or not. In this experiment, the analysis indicates that it is possible to confound the v_{net} ,C plot with that of Bovengton-Jones. Else more, this comparison permit to say that the Bovengton-Jones function is fundamentally based on the same theoretical considerations of the tow other functions. In this meaning, it is normal to think that this function may not fit the initial step of dissolution, unless this step being dominated by a conditions unfavorable to linearity. Then, the interpretation sometimes given in literature (Delmas,1975) to this function may be revised to say that the non adjustment of the initiation of dissolution by this function doesn't mean necessarily that the kinetic is not controlled by the chemical reaction.

CONCLUSION

Constituent of soil. If its dissolution or precipitation may be viewed as simple processes its dynamic in soil proceed however as a rather complex one. Due to the property of this component as a product of intermediate solubility its dissolution may be as fast as its precipitation. Even these two processes undergo reversibly from a thermodynamic viewpoint, the expansion of gypsum in situ behaves as a rather irreversible phenomenon simply because it is not easy to incur the dynamic of this component. A side the thermodynamic and hydric considerations, the kinetic aspect must be undertaken first since the transformations of gypsum in soil submitted to the irrigation cycles proceed out of equilibrium. Although the kinetic process of gypsum dissolution is simple in water, it may be somewhat complex in soils, especially in salt affected soils where the growth rate of gypsum may be

strongly affected in the presence of electrolytes Bosbach *et al* (1996). The kinetic laws applicable here may be more complex due to the ionic strength and to the secondary reactions which may take place. Under the effect of these reactions the kinetic may change its function with time, so it is likely, for a more exhaustive studies, to take this factor in consideration to see how the kinetic function does change with time and to make of this an extrapolation covering the irrigation cycles in soil.

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انحلال الجيبس "Fer de Lance" : دراسة حركية

علي عجيب

قسم التربة واستصلاح الأراضي، كلية الزراعة، جامعة تشرين، ص. ب ٢٠٨٠ اللاذقية، سورية.

استفاد هذا البحث من التسهيلات التي قدمها المعهد الوطني الفرنسي للبحوث الزراعية فـي مدينـة رين الفرنسية ضمن برنامج تعاون سوري– فرنسي مشترك حول الترب المالحة والجبسية في حوض الفرات السوري . تأتي أهميته من المكانة التي يحتلها الجيبس كمكون ترتبط به خواص التربة الفيزيائية والكيميائيـة حتى يطغى بصفاته وخواصه عندما تصبح نسبته في التربة فوق الحد المرغوب . مازال انحلالـه وترسبـه يثيران أكثر من تساؤل لاسيما عندما يتعلق الأمر بالسيطرة.على ديناميكيته في التربة وفي الطبقات التالية لها حيث ترتبط به في العديد من الحالات ظواهر الكارست وتشكل الكهوف الباطنية.

في هذه التجربة تمت دراسة بعض الجوانب الحركية لانحلال واحد من عـروق الجيـبس – " Fer de Lance" – في الماء وفي محلول من بيكربونات الصوديوم ٠,٠٠١ مولي. وكان ذلك بطحـن عينـة الجيبس وتعليق ٩ غرام من الشريحة الحبيبية 90-240 μm في ٥٠٠ ml من أحد المحلولين السابقين فـي حوجلة لها خلاط للتحريك ومنظم لدرجة الحرارة (25°C) ومأخذ لاعتيان المعلق. كان الحجم المأخوذ عند كل زمن من أزمنة القياس ٥ ml ، رشحت باستعمال رقائق ترشيح ملليبور TM . قـدر تركيـز الشاردتين Ca²⁺,SO²⁻ في الرشاحة بعدئذ بطريقة الكروماتوغرافيا الأيونية .

تظهر النتائج اختلاف الشاردتين فيما يتعلق بالكمية الذائبة من كل منهما، وتبين أن البيكربونات تعمـل على زيادة الانحلالية . من ناحية أخرى، يؤكد تحليل النتائج بتطبيق نظرية دافيس ونظرية الحالة الانتقاليـة على سلوك انحلال الجيبس باعتباره ظاهرة حركية من الدرجة الثانية تقع تحت تأثير التفاعل الكيميائي. كـان ذلك واضحاً فيما يتعلق بالانحلال في الماء لاسيما في المراحل التالية لبدئه حيث كـان التابـع بوفنكتـون – جونس Bovengton-Jones محققاً. إذا كانت بداية الانحلال لاتستجيب للتابع المذكورفقد لايكون فـي الأمر أكثر من تباين ظاهري يمكن أن يعزى، استناداً إلى البارامترات التي تم تعيينها بتطبيق نظرية الحالـة الانتقالية، إلى العلاقة غير الخطية بين سرعة الانحلال الشاملة V_{net} والطاقة الحرة للتفاعل الكيميائيΔG .

الكلمات المفتاح: الجيبس، الانحلال، حركية، طاقة التنشيط، نظرية الحالة الانتقاليـة، الانتشـار، التفاعـل الكيميائي، سرعة التفاعل.