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THE INFLUENCE OF PORE WATER ON THE COMPACTION BEHAVIOUR OF CARBONATE SAND

R.B. DE BOER[1] & J. LAGRAND[1]

ABSTRACT

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Maastrichtian Carbonate Sand collected from the Pietersberg near Maastricht (Netherlands) was compressed at room temperature, in order to detect pressure-solution phenomena. Compaction took place at slowly rising loads (10 atm/day) as well as under constant loading. The maximum loads to which the different samples were subjected varied from 15 to 280 atmospheres.

The influence of the following factors on the compaction behaviour of the sand was studied:

1. presence or absence of pore water;
2. partial CO_2-pressure of pore water;
3. inhibition of calcite precipitation;
4. saturation of pore water with respect to calcite;
5. circulation of pore water.

At room temperature applied microscopically detectable pressure-solution phenomena were not produced. The influence of the presence of pore water on compaction was clearly demonstrated, but the pore-water composition appeared not to be a dominant factor.

INTRODUCTION

The compaction behaviour of carbonate sand in the presence of a pore solution has been much less intensively studied by experiment than the compaction behaviour of quartz under this condition. Griggs (1940) deformed Yule Marble and Solnhofen Limestone and found that the presence of water induced a transition from brittle to ductile behaviour of these materials. Yule Marble could be shortened by 40% at 150°C under a differential stress of only 1 500 kg/cm^2 when carbonated water was present in the pores. Deformation of a dry sample called for a differential stress of at least 8 200 kg/cm^2. Contrary to the sample deformed in dry condition, the sample deformed in the presence of water did not exhibit increased formation of gliding planes or twinning. It must therefore be assumed that these deformation indicators were either not formed, and that pressure solution occurred instead, or that recrystallisation removed any traces during or shortly after the experiments. Griggs did not specify the deformation rate applied in this compaction experiment, but from his experimental data on other specimens it may be concluded that all deformations in the presence of water were carried out at extremely low rates.

Experiments carried out by Rutter (1972, 1974) on Solnhofen Limestone, in contrast, revealed no sign of pressure solution or recrystallisation. In Rutter's experiments the influence of pore water as found by Griggs was entirely absent or only very small. It should be mentioned that, unlike Griggs' experiments, most of Rutter's were of short duration (although in some of them the straining rate was as low as 3×10^{-8}/s).

In studying the influence of pore water on rock strength it is essential that very low loading rates be applied to the sample (Lowry, 1956), in order to avoid purely mechanical deformation processes becoming so intensive as to disguise the effects of other mechanisms that may be operative at the extremely low straining rates that occur in nature. The experiments carried out by Griggs are therefore considered more relevant for studying the compaction process in nature than Rutter's, especially in the diagenetic realm.

Compaction experiments on unconsolidated carbonates

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[1] Koninklijke/Shell Exploratie en Produktie Laboratorium, Rijswijk, The Netherlands.

were carried out by Fruth, Orme & Donath (1966) and by Ebhardt (1968). Ebhardt noted a difference in compaction behaviour between carbonate sands containing air, benzene and water in the pores. Unfortunately, he was unable to relate any of his results to natural diagenetic processes.

The data obtained on carbonate compaction are in contrast with those found for quartz. For quartz it was reported that the presence of pore water promotes plastic flow (Griggs, 1967) and fracturing (Swolfs, 1972). Experiments on quartz sand were carried out by Maxwell (1960), who concluded that in his experiments consolidation took place by straining, fracturing and crushing of the grains, followed by dissolution. It is, however, very doubtful whether these experimental results are applicable to natural conditions, because Maxwell loaded his samples in one step instead of gradually (Lowry, 1956). Pressure-solution phenomena were observed in experimentally compacted quartz sand by Renton, Heald & Cecil (1969) at 400°C and by de Boer (1975) at 340°C, who both applied very low loading rates (5 – 15 atm/day).

In a study on pressure-solution phenomena in carbonate sands, carried out in Shell's Rijswijk laboratories, these sands were compacted at room temperature, after which the influence of the following factors was studied: the presence of pore water, its degree of saturation with respect to calcite and its partial CO_2-pressure, Mg^{++}-content and flushing rate. Although not always identical carbonate samples were used for the various experiments, the emphasis was in all cases on the nature of the pore fluid and not on that of the solid phase.

METHODS AND MATERIALS

Carbonates have two characteristics that make them extremely suitable for measuring the influence of pore water on compaction behaviour, viz:

1. At low temperatures carbonate minerals are more soluble than quartz. Hence, there is a possibility of finding pressure-solution phenomena at moderate to low temperatures.
2. It is relatively easy to change the concentrations of the components of the carbonate-water-CO_2 equilibrium: Ca^{++}, $CO_3^=$, HCO_3^-, CO_2, H^+, OH^- etc. It was hoped that by varying these conditions an impression could be gained of the compaction mechanism.

A disadvantage could be that calcite precipitation is strongly inhibited by impurities such as Mg^{++} (de Groot & Duyvis, 1966), Fe^{++} and by many organic impurities (Suess, 1970), but at the time of the investigation this was thought to be of minor importance.

Used as compaction material were different sieve fractions of the Maastrichtian chalk from the Pietersberg near Maastricht (The Netherlands), the properties of which are as follows:

1. The material is very friable and only slightly cemented.
2. It is purely calcitic, so no interference with recrystallisation (aragonite → calcite) is to be feared. This recrystallisation might cause appreciable compaction of loaded, porous material.
3. The chalk is full of small fossils (echinoderms) on which pressure solution should be easily observable.

After the pores had been filled with a saturated calcium carbonate solution, the chalk was compacted in a uniaxial compaction apparatus as described by van der Knaap & van der Vlis (1967). The load on the grains was increased very slowly at a maximum rate of 10 atm/day, while the pore fluid was kept at 1 atm (loading gradually with small increments prevents extreme grain breakage and brings out the effects of slow recrystallisation processes).

EXPERIMENTS

The compaction behaviour of the chalk material was studied both under increasing loads and at constant loading. In the case of increasing loads there was very little difference in compaction behaviour between the various sieve fractions and no influence of pore-water composition could be observed. When subjected to constant loading, the samples showed gradual compaction at a constantly decreasing rate, which can be described by the formula

$$\frac{\Delta\varphi}{\varphi_t} = K . \ln t + K'$$

where φ_t = porosity at time t (expressed in hours)

$\Delta\varphi$ = $\varphi_i - \varphi_t$

t = O at onset of constant loading

φ_i = initial porosity (at zero load)

K = constant; hereinafter referred to as 'compaction factor'

K′ = $(\varphi_i - \varphi_1)/\varphi_1$ (where $\varphi_1 = \varphi_t$ at t = 1)

This formula is valid as from one hour after the load has been kept constant (see Figs. 1, 3, 4, 6 and 7). The compaction factor K has a value of the order of 6×10^{-3}, so that compaction for the first 400 hours resulted in a porosity decrease of approx. 1% only.

As appears from Table I, it was necessary to allow for a variation in φ_1 of 5%, and a variation in compaction constant of a factor of 2.

The experiments showed an unwanted interference by the size of the compaction cell, which must be due to cell-wall friction.

No difference was observed between the experiments with pore water saturated with pure CO_2 at 1 atm. and those with water saturated with air, in which the partial CO_2 pressure is only 0.0004. Lowering the partial CO_2 pressure by this factor will reduce the calcite solubility by a factor of 16, and

Table I.
Compaction behaviour of Maastrichtian chalk.

Exp. No.	Load (atm)	P_{CO_2} (atm)	φ_1 (%)	K[1] x10^3	Sieve Fraction (mesh)	Sample size ϕ (mm)
2	50	1	41	8.1	10-20	50
3	80	1	36	6.8	10-20	50
4	15.5	0.0003	53	6.4	10-20	50
5	15.6	0.0003	52	3.4	10-20[2]	50
4a	50	0.0003	44	8.7	10-20	50
5a	50	0.0003	45	3.5	10-20[2]	50
1	60	1	41	0.95	7-20	35
8	60	1	43	2.0	7-20	35
7	60	1	39	4.2	20-50	35
6	60	1	44	2.4	50-200	35
11	60	1	49	2.5	50-200	35
9	60	0.0003	47	2.9	50-200	35
12	90	0.0003	43	3.9	50-200	35
13	90	0.0003	45	5.0	50-200	35
14	90	0.0003	45	2.8	50-200	35
15	120	0.0003	43	4.4	50-200	35
17	162	0.0003	40	6.9	50-200	35
17d	280	0.0003	37	7.8	50-200	35
17a	162	(iso-octane)	39	0.22	50-200	35
17b	215	(iso-octane)	39	0.39	50-200	35
17c	280	(iso-octane)	37	3.5	50-200	35

[1] K from equation 1
[2] Sample in exp. 5 different from that used in exp. 4.

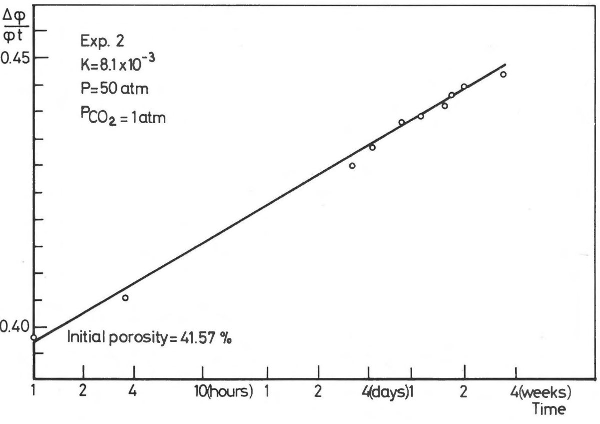

Fig. 1
Compaction behaviour of carbonate sand under constant load.

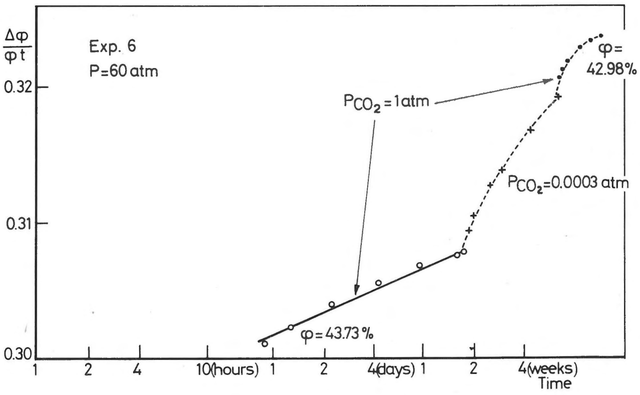

Fig. 2
Influence of mixing pore solutions of different composition.

the apparent diffusion coefficient of calcite by a factor of 200 (de Boer, 1975, Appendix II).

To ensure that CO_2 pressure had no real influence, it was decided to exchange the pore solutions during compaction, so that changes in K-value could easily be seen. However, both replacement of a high-CO_2 solution by a low-CO_2 solution and the reverse resulted in extra compaction (Fig. 2). This is probably due to the fact that mixing of the two solutions, both saturated with respect to calcite, yields an undersaturated solution. Apparently, dissolution occurred during the exchange and this resulted in extra compaction.

In experiment 11 and subsequent experiments the samples were first dried before a new solution was allowed to flush in, so that no mixing could take place. Changing from a high-CO_2 to a low-CO_2 solution had no influence on the compaction constant (Fig. 3). However, the reverse exchange again resulted in extra compaction, in spite of the precautions taken. This is possibly due to the fact that the injected water was still somewhat undersaturated with respect to the stressed grains.

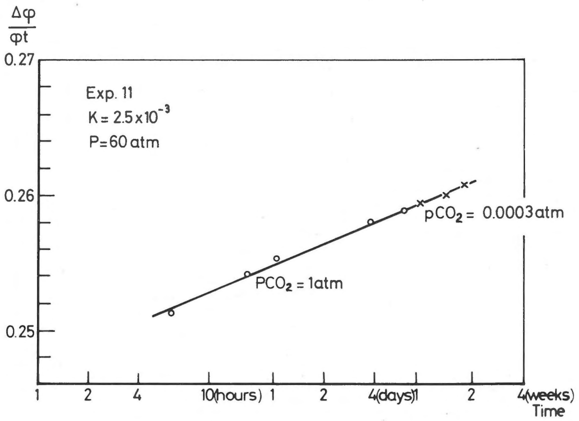

Fig. 3
Influence of pore-water composition on compaction of carbonate sand.

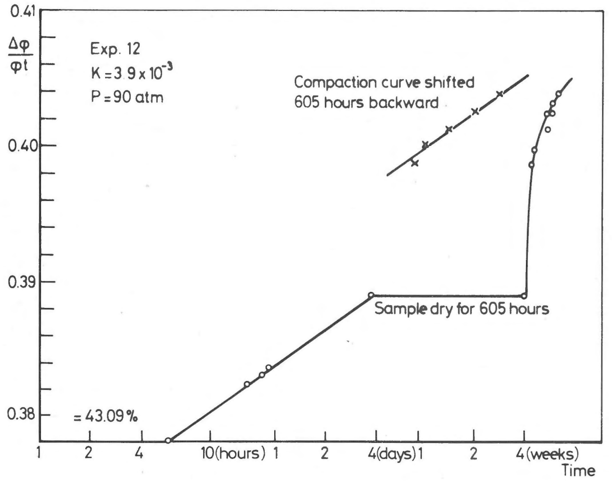

Fig. 4
Influence of presence of pore water on compaction.

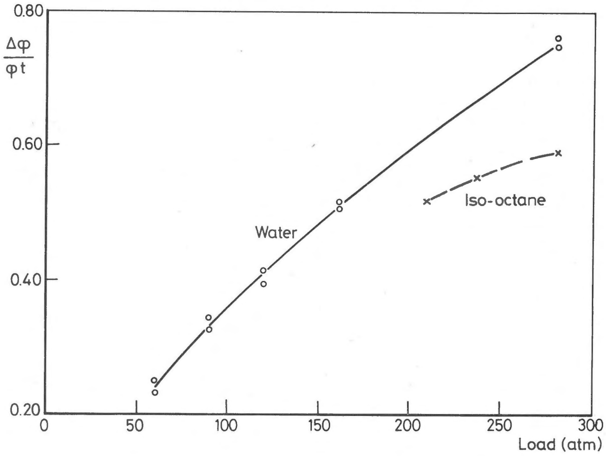

Fig. 5
Compaction behaviour of carbonate sand in the presence of water and iso-octane under increasing load.

The obvious indifference of the compaction behaviour with respect to the pore-fluid composition was the incentive to carry out experiment No. 12 to investigate whether water had any influence at all. After a normal compaction test, the pore solution was removed and the sample dried. No compaction occurred in the following 605 hours, until water was re-injected. Then the sample compacted rapidly after which compaction went on as if time had stopped for the previous 605 hours (Fig. 4). An even clearer example showing the special influence of water is experiment No. 17, induced by a finding of Ebhardt (1968). Ebhardt had observed that when benzene was used as pore fluid, the samples were much more compressible than when using water (this may be explained by the much higher initial porosity of the benzene sample). He ascribed this phenomenon to the lubricating effect of benzene. To test this effect at constant loading, a sample was loaded up to 165 atm in low-CO_2 water and the compaction factor was determined over the next five hours, after which pore water was replaced by iso-octane. The compaction factor immediately dropped to near zero (Table I). A load increase to 215 atm in iso-octane led to a much lower compaction than occurred in the presence of pore water (Fig. 5). Moreover, this compaction was not smooth, as normally observed, but proceeded with small shocks, apparently resulting from a 'cascade' of grain breakages. At this pressure, the compaction at constant loading was negligible again (exp. 17b). Only after a load increase to 280 atm did the sample show reasonable compaction at constant loading. When the pore water was re-injected, compaction proceeded with the same K-factor as the start of the experiment (exp. 17d). These phemomena indicate the considerable influence of the chemical properties of water on the compaction behaviour of carbonates. From Fig. 2 it is apparent that the solubility of the carbonate in the pore fluid is an essential factor. For slightly soluble minerals such as calcite, no dissolution can occur without recrystallisation, so we tried to stop precipitation to see how compaction would react to this.

After a normal compaction experiment, the pore water was removed, the sample dried, and the dry sample immersed in a Mg^{++}-rich solution, saturated with calcite. As the Mg^{++} -ion strongly inhibits calcite precipitation (de Groot & Duyvis, 1966), we expected the compaction to slow down immediately. The reverse took place, however; the compaction factor showed a slight increase instead of a decrease. This increase is not significant, because after a renewed influx of the original Mg^{++}-free pore water the compaction factor again showed a slight increase. It can therefore be concluded that inhibition of calcite precipitation had no effect on compaction behaviour (Fig. 6).

Another factor that might be of importance is the rate of transport of dissolved material to places where it can precipi-

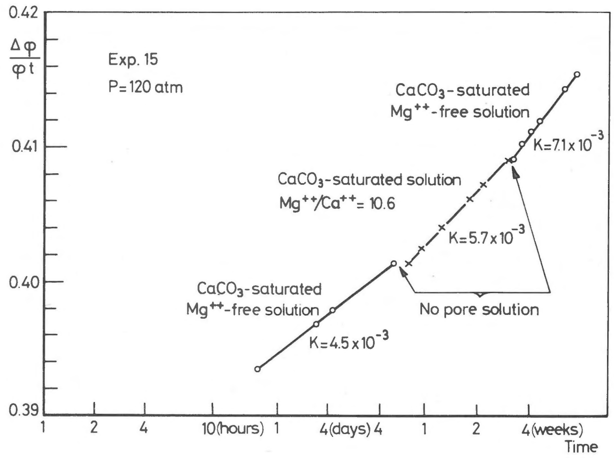

Fig. 6
Influence of Mg^{++} ions on compaction behaviour of carbonate sand.

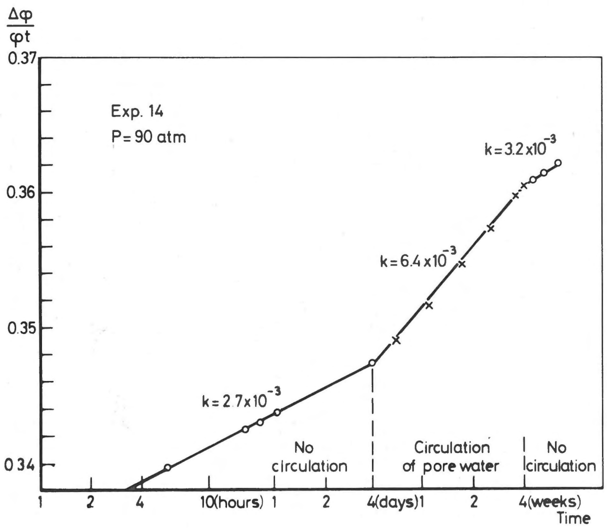

Fig. 7
Influence of pore-water circulation on compaction behaviour.

tate. Movement of the pore water might therefore influence compaction rate. This was tested in experiments 10 and 14. In experiment 10, after normal compaction, calcite-saturated pore water was flushed through at different rates. The compaction factor increased when the flushing rate increased and decreased when flushing was stopped again. This effect could still be due to slight undersaturation of the pore water with respect to the stressed grains. In experiment 14 the pore water was therefore circulated. The flushing rate was 2 ml/h and the total quantity of circulating fluid was 60 ml, so that the fluid circulated once every 30 hours. In the 20 days during which circulation went on, the compaction factor was constantly higher than under stagnant conditions and dropped to its original value as soon as circulation was stopped (Fig. 7).

CONCLUSIONS

From these experiments we learned the following:

1. The compaction behaviour of this Maastrichtian chalk is, neither at increasing loads nor under constant loading, demonstrably influenced by pore-water composition, when the pore water is saturated with chalk.
2. Compaction under constant loading is very small, compared with compaction at increasing loads. This conclusion can also be derived from the work of Fruth, Orme & Donath (1966) and of Ebhardt (1968).
3. The compaction rate increases appreciably when pore water is undersaturated.
4. Compaction stops when during constant loading pore water is replaced by a fluid in which calcite is insoluble.
5. Inhibition of calcite precipitation does not affect compaction behaviour.
6. Movement of pore fluid enhances compaction.
7. For this Maastrichtian chalk the compaction behaviour can be expressed by:

$$\frac{\Delta\varphi}{\varphi_t} = K.\ln t + K'$$

In the thin sections studied it was observed that many grains showed breakage, especially in the coarser fractions; no overgrowth was observed that could with certainty be regarded as having been formed during the experiment. Intergrowth of grains was difficult to observe, because all particle boundaries were blurred by a micritic rim, which readily collapsed at contacts between particles. Thin section study of these samples is therefore a very uneasy route to gain insight into the various phenomena.

DISCUSSION

Interpretation of the results leaves some questions. It is difficult to explain why both the presence of water and its degree of (under) saturation have such a pronounced influence, in contrast with the composition of the water if saturated with respect to calcite. One could argue that the absence of an influence of Mg^{++} is related to aragonite precipitation not being inhibited. Aragonite has a solubility product of only 1.5 times that of calcite, but the influence of stress on the solubility of calcite is too low to neglect this difference in solubility. To reach the solubility product of aragonite, a calcite crystal must be subjected partly or wholly to a stress of 330 atm. Thus, when calcite precipitation is inhibited by addition of Mg^{++}, all calcite surfaces subject to a stress of less than 330 atm will cease contributing to the pressure-solution process. Calcite surfaces exposed to a stress of more than 330 atm may proceed dissolving, although at lower rates, because they now have to recrystallise to a mineral with a higher solubility. Therefore, since no influence of Mg^{++}-addition can be observed, the actual stresses must have been an order of magnitude higher than 330 atm.

Acceptance of the usual mechanism for pressure solution (Weyl, 1959) as a cause of the observed compaction process also leaves the absence of the influence of CO_2-content unexplained. Only one conclusion seems reasonable: in spite of the low loading rates, the dominant reaction is not pressure solution, but the mechanism mentioned by Maxwell (1960) 'mechanical straining, fracturing and crushing at points of contact, followed by dissolution'. In these cases the presence of water is essential for continuance of the reaction, but the kinetics can be completely determined by purely mechanical reactions. Only undersaturation of pore water results in dissolution, and hence in enhanced compaction. However, such an explanation neglects the influence of pore-water circulation. One could speculate about a mechanism of which the rate-determining factors are the mechanical reaction and the diffusion of the dissolved material to the places of lower stress, but it seems advisable to leave this question open until more data have been collected. In any case the experiments conducted have demonstrated the pronounced influence of the presence of pore water and its saturation on the compaction behaviour of carbonate sand.

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