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***T.J. Katsube, N. Scromeda, and S. Connell***

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# Thicknesses of adsorbed water layers on sediments from the JAPEX/JNOC/GSC Mallik 2L-38 gas hydrate research well, Northwest Territories

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**Abstract:** Thicknesses and number of water molecular layers adsorbed on pore surfaces have been determined for 11 sedimentary rocks from 850–950 m depths in the Mackenzie Delta (Northwest Territories), using vacuum drying at temperatures of 4–100°C. The purpose was to determine adsorbed water characteristics of unconsolidated to moderately consolidated sediments, as part of a fluid expulsion and entrapment study of compacting sediments. Whereas free water is evacuated under vacuum, about five adsorbed water layers are known to exist on clay surfaces that can not be evacuated under those conditions at less than 25°C.

These results indicate that there are generally 0–3 adsorbed water layers for naturally and vacuum dried samples at 25°C; and larger numbers, 7–270 and 1–70, for samples naturally dried at 4°C and vacuum dried at 25°C, respectively. In addition, unlike previous cases most adsorbed water is evacuated at 50°C, for these samples.

**Résumé :** L'épaisseur et le nombre de couches moléculaires d'eau adsorbées sur les surfaces des pores de 11 échantillons de roches sédimentaires, prélevés à des profondeurs de 850 à 950 m dans le delta du Mackenzie (Territoires du Nord-Ouest), ont été déterminées par séchage sous vide à des températures de 4 à 100 °C. Ces mesures visaient à déterminer les caractéristiques de l'eau adsorbée des sédiments non consolidés à modérément consolidés dans le cadre d'une étude de l'expulsion et du piégeage des fluides dans des sédiments en voie de compaction. Bien que l'eau libre soit évacuée sous vide, on sait qu'il existe environ cinq couches d'eau adsorbée sur les surfaces d'argile qui ne peuvent être évacuées dans ces conditions à moins de 25 °C.

Ces résultats indiquent qu'il y a généralement de 0 à 3 couches d'eau adsorbée pour les échantillons séchés à l'air libre et sous vide à 25 °C, et de plus grands nombres, de 7 à 270 et de 1 à 70, respectivement, pour les échantillons séchés à l'air libre à 4 °C et sous vide à 25 °C. En outre, à l'inverse de ce qui avait été observé dans les cas précédents, la plus grande partie de l'eau adsorbée est évacuée à 50 °C dans le cas de ces échantillons.

## INTRODUCTION

The adsorbed water thicknesses and number water molecular layers (double-layers) on pore surfaces have been determined for 11 sedimentary rock samples from 850–950 m in the Mallik 2L-38 well (Northwest Territories, Dallimore et al., (1999)). The purpose was to obtain information on the adsorbed water characteristics of unconsolidated to moderately consolidated sediments, as part of a fluid expulsion and entrapment study of compacting sedimentary material. Information on adsorbed water characteristics, in the form of ‘static irreducible water saturation’, has been reported on a routine basis for various types of rocks as part of the immersion porosity determination procedure (e.g. Scromeda and Katsube, 1994). The data used in this study was produced by a previous study of this nature for the Mallik 2L-38 well sediments (Scromeda et al., 1999).

The moisture of moist rocks consists of free water molecules and water molecules adsorbed to the grain surfaces. When the rock is dried under vacuum or naturally dried at temperatures below 100°C, only the free water content is evacuated, and the adsorbed water remains on the grain surfaces (e.g. Hinch, 1980); however, the amount of adsorbed water decreases with increased temperature, with all adsorbed water evacuated at 116°C (e.g. Hinch, 1980). Another purpose of this study was to determine if the adsorbed water content shows an increase with decreased temperature below 25°C. This paper reports the results of analyses performed on data that has been obtained at temperatures of 4°C, 25°C, and 50°C. In this paper, in addition to a brief description of the procedures used to obtain the adsorbed water data, the methods used to determine the adsorbed water thicknesses and their results are presented.

## THEORY

If we assume that a sample of sedimentary material has a dry volume and dry weight of  $V_D$  and  $W_D$ , respectively, then its bulk density,  $\delta_D$ , would be expressed by

$$\delta_D = W_D/V_D. \quad (1)$$

If the pore-surface area and adsorbed water layer thickness of the sample are  $A$  and  $d_p$ , respectively, then we can assume that the adsorbed water layer volume,  $V_L$ , and weight,  $W_L$  are expressed by

$$V_L = (AW_D)d_p, \quad (2)$$

and

$$W_L = V_L \delta_W, \quad (3)$$

respectively, where  $\delta_W$  is the bulk density of water. The units of  $A$  are in square metres per gram ( $m^2/g$ ), so that  $AW_D$  (Equation 2) represents the actual surface area of the sample that has a weight of  $W_D$  (in grams).

A vacuum-dried sample is considered to be absent of free water. Since, in this case, the adsorbed water layers would be the only addition to the dry material, its wet volume,  $V_W$ , and wet weight,  $W_W$ , would be

$$V_W = V_D + V_L \quad (4)$$

and

$$W_W = W_D + W_L \quad (5)$$

respectively. Therefore, if we know  $W_W$ ,  $W_D$ , and  $A$  of a sample, then from Equations (5), (3), and (2), we can derive the thickness of the adsorbed water layers,  $d_S$ :

$$d_S = V_L/(AW_D) = W_L/\delta_W/(AW_D) = (W_W - W_D)/(AW_D \delta_W). \quad (6)$$

We consider the density of water ( $\delta_W$ ) to be unity.

It should be noted that the concept expressed in Equation (4), actually, applies only to powder or loose material, where the individual grains of the sample are coated with adsorbed water layers, and the volume of the wet sample could be reasonably larger than that of the dry sample. In a solid rock sample,  $V_L$  may represent only the adsorbed water on the outer surface of the solid sample and not that on the pore surfaces (e.g. low porosity rocks). That is,  $V_L$  would be negligible and in this case

$$V_W = V_D. \quad (7)$$

## ANALYTICAL APPROACH

### Samples and data

Static irreducible saturation data have been reported for 17 sediment samples (Scromeda et al., 1999) from the core material of the Mallik 2L-38 well located in the Mackenzie Delta, Northwest Territories. The data for 11 of these samples have been selected for this study. Information on sample depth and lithology are listed in Table 1.

Part of the sample preparation procedure, previously described (Scromeda et al., 1999), are repeated here. The specimens used for the original testing were taken from subsamples which had been stored first at low temperatures (4°C) and then at room temperature. Two specimens (A and B) were initially taken from each of the unconsolidated to

**Table 1.** Information on sample depth and lithology (Jenner et al., 1999).

Sample number	Depth (m)	Lithology
EJA-1	896.8	Silt
EJA-2	896.4	Clayey silt
EJA-3	887.3	Silt
EJA-4	897.1	Sandy silt
EJA-5	906.2	Sandy
EJA-6	919.1	Sand
EJA-8	927.6	Clayey silt
EJA-9	949.4	Clayey silt
EJA-13	903.9	Silt, clayey silt
EJA-14	936.7	Clayey silt
EJA-15	944.5	Clayey silt

moderately consolidated subsamples. They were then placed in a weighed beaker at room temperature (25°C). The specimens were saturated with 5–10 mL of deionized water in a beaker, just enough to fully cover the specimens. The A specimens were placed in room atmosphere and temperature (25°C) while the B specimens were placed in a refrigerator at 4°C. Both the A- and B-series specimens remained at these temperatures, until all of the excess water evaporated out of the beakers, leaving only the moist specimens. Their wet weights,  $W_W$ , were determined at this time. All specimens were then placed in a vacuum chamber for initial natural drying (Katsube and Scromeda, 1995). The vacuum was then applied at 760 mm Hg for 420 minutes at room temperature (25°C), until a constant weight,  $W_{V25}$ , was reached. Following this procedure, the specimens were placed in the vacuum-oven under a vacuum of 760 mm Hg and dried for 24 hours at two different temperatures: 50°C and 100°C. Weights,  $W_{V50}$  and  $W_D$ , were measured after constant weights were reached at 50°C and subsequently at 100°C, respectively. The  $W_D$  is the constant weight at 100°C, and is considered to represent the sample when it is devoid of any free and adsorbed

(or bound) water (American Petroleum Institute, 1960). These drying procedures are described in further detail elsewhere (Scromeda and Katsube, 1993; Scromeda et al., 1999). These data ( $W_W$ ,  $W_{V25}$ ,  $W_{V50}$ , and  $W_D$ ) for both the A and B series of the 11 samples are listed in Tables 2a and 2b. The bulk density,  $\delta_D$ , and surface area,  $A$ , values for the samples that are listed in these tables were obtained from the literature (Katsube et al., 1999). The surface area values were obtained by mercury porosimetry.

### Analytical procedure

The adsorbed water thickness ( $d_S$ ) values are derived from the surface area ( $A$ ) and specimen weight data ( $W_W$ ,  $W_{V25}$ ,  $W_{V50}$  and  $W_D$ ) listed in Tables 2a and 2b, using Equation (6). The parameter for  $W_W$  in Equation (6) was replaced by  $W_{V25}$  and  $W_{V50}$  when deriving the adsorbed water thicknesses for vacuum dried at room (25°C) and 50°C temperatures:  $d_{S25}$  and  $d_{S50}$ , respectively. The adsorbed water thickness of the wet specimen at room temperature is represented by  $d_{SW}$ .

**Table 2a.** Adsorbed water layer characteristics for 11 mudstone samples saturated and naturally dried at room temperature (25°C). The samples in this table are represented by the “a” specimens (e.g. EJA-1a, EJA-2a .....EJA-15a).

Sample	h (m)	$\delta_D$ (g/mL)	A (m <sup>2</sup> /g)	$W_W$ (g)	$W_{V25}$ (g)	$W_{V50}$ (g)	$W_D$ (g)	$d_{SW}$ (nm)	$d_{S25}$ (nm)	$d_{S50}$ (nm)	$n_{SW}$	$n_{S25}$	$n_{S50}$
EJA-1a	896.8	2.73	14.8	0.8702	0.8695	0.8620	0.8611	0.714	0.659	0.071	2.64	2.44	0.26
EJA-2a	896.4	2.66	20.9	0.5398	0.5391	0.5356	0.5355	0.384	0.322	0.009	1.42	1.19	0.03
EJA-3a	887.3	2.65	10.9	1.3733	1.3726	1.3662	1.3658	0.504	0.457	0.027	1.87	1.69	0.1
EJA-4a	897.1	2.65	14.2	1.4195	1.4190	1.4094	1.4092	0.515	0.490	0.010	1.91	1.81	0.04
EJA-5a	906.2	2.66	13.9	0.8010	0.8007	0.7993	0.7991	0.171	0.144	0.018	0.63	0.53	0.07
EJA-6a	919.1	2.65	14.4	0.7032	0.7029	0.7022	0.7021	0.109	0.0791	0.010	0.4	0.29	0.04
EJA-8a	927.6	2.63	14	1.4942	1.4936	1.4838	1.4835	0.515	0.486	0.014	1.91	1.8	0.05
EJA-9a	949.4	2.65	20.4	1.1605	1.1598	1.1546	1.1542	0.268	0.238	0.017	0.99	0.88	0.06
EJA-13a	903.9	2.60	18.1	2.3518	1.7919	1.3907	1.3890	38.3	16.0	0.068	142	59.4	0.25
EJA-14a	936.7	2.52	13.1	2.9682	2.1568	1.6140	1.6127	64.2	25.8	0.062	238	95.4	0.23
EJA-15a	944.5	2.57	16.6	3.9536	3.4149	3.0408	3.0356	18.2	7.53	0.103	67.5	27.9	0.38

h= Depth of sample  
 $\delta_D$ = Bulk density of sample  
A= Pore surface area  
 $W_W$ = Weight of sample naturally dried and weighed at room temperature (25°C)  
 $W_{V25}$ = Vacuum-dried weight of sample at 25°C  
 $W_{V50}$ = Vacuum-oven dried weight of sample at 50°C  
 $W_D$ = Vacuum-oven dried weight of sample at 100°C  
 $d_{SW}$ = Thickness of adsorbed water layer of wet sample at room temperature (25°C)  
 $d_{S25}$ = Thickness of adsorbed water layer at 25°C  
 $d_{S50}$ = Thickness of adsorbed water layer at 50°C  
 $n_{SW}$ = Number of adsorbed water layers for wet sample at room temperature (25°C)  
 $n_{S25}$ = Number of adsorbed water layers at 25°C  
 $n_{S50}$ = Number of adsorbed water layer at 50°C

Estimated experimental error ranges:  
<±10%:  $\delta_D$ ,  $W_W$ ,  $W_{V25}$ ,  $W_{V50}$ ,  $W_D$   
± 10–20%: A.  
± 10–40%:  $d_{SW}$ ,  $d_{S25}$ ,  $d_{S50}$ ,  $n_{SW}$ ,  $n_{S25}$ ,  $n_{S50}$

## ANALYTICAL RESULTS

The results of the adsorbed layer thicknesses for the sets of samples naturally dried at room temperature (25°C) and at 4°C are listed in Table 2a and 2b, respectively. The adsorbed water thicknesses ( $d_s$ ) determined for the samples at the wet, vacuum dried at room (25°C) and 50°C temperatures are represented by  $d_{SW}$ ,  $d_{S25}$ , and  $d_{S50}$ , respectively, in these two tables.

The results for  $d_{SW}$  and  $d_{S25}$  show little differences, for the set of samples naturally dried at room temperature (25°C, Table 2a), and are in the range of 0.1–0.71 nm, for most of these samples. Three samples (EJA-13, EJA-14, EJA-15) show values of 7.5–64 nm, and are an anomaly in this set of measurements. The results for  $d_{S50}$  are in the range of 0–0.1 nm for all 11 samples. The number of adsorbed water molecular layers,  $n_{SW}$ ,  $n_{S25}$ , and  $n_{S50}$ , for wet, vacuum dried at room (25°C), and at 50°C temperatures were determined by dividing the  $d_{SW}$ ,  $d_{S25}$ , and  $d_{S50}$  values by 0.27 nm. This is based on the assumption that a water molecule, under these conditions, has a diameter of 0.27 nm (Hinch, 1980). The  $n_{SW}$  and  $n_{S25}$  values are in the range of 0–3 layers for most of the

samples, and 28–240 for the three anomalous samples (EJA-13, EJA-14, EJA-15). The values for  $n_{S50}$  are all less than 0.4, and are interpreted to be zero.

The results of  $d_{SW}$ ,  $d_{S25}$ , and  $d_{S50}$  for the set of samples naturally dried at 4°C (Table 2b) show considerable difference with those naturally dried at room temperature (25°C). The  $d_{SW}$ ,  $d_{S25}$  and  $d_{S50}$  values for this set of samples are in the ranges of 1–56, 0.2–20, and 0–0.25 nm, respectively. The corresponding number of adsorbed water molecular layers,  $n_{SW}$ ,  $n_{S25}$ , and  $n_{S50}$  are in the ranges of 5–210, 0.6–75, and 0–1 layers.

## DISCUSSION AND CONCLUSIONS

Whereas free water can be evacuated under vacuum conditions, previous studies (e.g. Hinch, 1980) indicate that there are about five strongly adsorbed water layers on surfaces of clay particles that can not be evacuated under those conditions at room temperature or below (<25°C). Our numbers ( $n_{SW}$  and  $n_{S25}$ ) of 0–3 layers for naturally and vacuum dried samples at room temperature (25°C) conditions are smaller than the reported results, except for the anomalous samples

**Table 2b.** Adsorbed water layer characteristics for 11 mudstone samples saturated and naturally dried at 4°C. The samples in this table are represented by the “b” specimens (e.g. EJA-1b, EJA-2b .....EJA-15b).

Sample	h (m)	$\delta_D$ (g/mL)	A (m <sup>2</sup> /g)	$W_w$ (g)	$W_{V25}$ (g)	$W_{V50}$ (g)	$W_D$ (g)	$d_{SW}$ (nm)	$d_{S25}$ (nm)	$d_{S50}$ (nm)	$n_{SW}$	$n_{S25}$	$n_{S50}$
EJA-1b	896.8	2.73	14.8	2.1057	1.4722	1.1540	1.1510	56.0	18.9	0.176	208	69.8	0.65
EJA-2b	896.4	2.66	20.9	0.5694	0.5655	0.5450	0.5438	2.25	1.91	0.105	8.34	7.07	0.39
EJA-3b	887.3	2.65	10.9	1.7893	1.5528	1.5104	1.5080	17.1	2.73	0.146	63.4	10.1	0.54
EJA-4b	897.1	2.65	14.2	1.5879	1.2039	1.1746	1.1736	24.9	1.82	0.060	92.1	6.73	0.22
EJA-5b	906.2	2.66	13.9	0.9300	0.7862	0.7846	0.7845	13.3	0.156	0.009	49.4	0.58	0.03
EJA-6b	919.1	2.65	14.4	0.6508	0.6420	0.6396	0.6396	1.21	0.261	0	4.5	0.97	0
EJA-8b	927.6	2.63	14.0	1.6356	1.5127	1.4565	1.4516	9.05	3.01	0.240	33.5	11.1	0.89
EJA-9b	949.4	2.65	20.4	1.1251	1.1195	1.0846	1.0822	1.94	1.69	0.109	7.2	6.26	0.4
EJA-13b	903.9	2.60	18.1	1.7915	1.3311	0.9732	0.9753	46.2	20.2	0.164	171	74.6	0.61
EJA-14b	936.7	2.52	13.1	2.8959	2.5052	2.4355	2.4319	14.6	2.30	0.113	53.9	8.52	0.42
EJA-15b	944.5	2.57	16.6	3.2469	2.8752	2.6895	2.6783	12.8	4.43	0.251	47.4	16.4	0.93

h= Depth of sample  
 $\delta_D$ = Bulk density of sample  
 A= Pore surface area  
 $W_w$ = Weight of sample naturally dried and weighed at room temperature (25°C)  
 $W_{V25}$ = Vacuum-dried weight of sample at 25°C  
 $W_{V50}$ = Vacuum-oven dried weight of sample at 50°C  
 $W_D$ = Vacuum-oven dried weight of sample at 100°C  
 $d_{SW}$ = Thickness of adsorbed water layer of wet sample at room temperature (25°C)  
 $d_{S25}$ = Thickness of adsorbed water layer at 25°C  
 $d_{S50}$ = Thickness of adsorbed water layer at 50°C  
 $n_{SW}$ = Number of adsorbed water layers for wet sample at room temperature (25°C)  
 $n_{S25}$ = Number of adsorbed water layers at 25°C  
 $n_{S50}$ = Number of adsorbed water layer at 50°C

Estimated experimental error ranges:  
 <±10%:  $\delta_D$ ,  $W_w$ ,  $W_{V25}$ ,  $W_{V50}$ ,  $W_D$   
 ±10–20%: A  
 ±10–40%:  $d_{SW}$ ,  $d_{S25}$ ,  $d_{S50}$ ,  $n_{SW}$ ,  $n_{S25}$ ,  $n_{S50}$

(EJA-13, EJA-14, EJA-15) that had 28–240 layers. The reason for this discrepancy is not yet understood. One consideration for the lower values is the adequacy of the surface area values (A) that were used (Equation 6). Another is the type of sedimentary material varies from sand and/or silt-rich to clayey mudstone (Table 1). The five strongly adsorbed water layers referred to in the literature (Hinch, 1980) are for pure clay material. No suggestions can currently be found to explain the reason for the larger numbers of adsorbed water layers for the anomalous samples.

The A values used in this study are in the range of 13–21 m<sup>2</sup>/g (Tables 2a, 2b), and were determined by mercury porosimetry. According to the current method used for their determination (Katsube et al., 1997), all porosity including storage porosity is treated as connecting porosity. Since these two types of porosities are about equal in most shale and mudstone (Katsube et al., 1997, 1998), this would imply that these A values are excessive by a factor of about two. If assumed that this were correct, the number of adsorbed water layers for the majority of our samples discussed above would double to 0–6 layers and be more or less in accordance to the literature values. On the other hand, it is well known that the mercury does not penetrate pores smaller than 3 nm (Katsube and Issler, 1993) by the porosimetry method currently used for these measurements, and that a considerable amount of surface area could be missed (Katsube, 1992). Therefore conclusive results on the adequacy of the A values used in these studies will have to wait for completion of a shale-mudstone surface area study currently underway.

The number of adsorbed water molecular layers ( $n_{SW}$  and  $n_{S25}$ ) for the naturally dried at 4°C and the vacuum dried at room temperature (25°C) conditions are 7–210 and 0.6–70, respectively (Table 2b). These values are considerably higher than those at corresponding conditions for the samples naturally dried at room temperature (Table 2a). In addition, the magnitude of these values vary considerably, with the three anomalous samples (EJA-13, EJA-14, EJA-15), at room temperature, no longer appearing as anomalous. Since the estimated error ranges of these  $n_{SW}$  and  $n_{S25}$  values are within 10–40% (Tables 2a, 2b), these comparisons are considered to be highly reliable.

Whereas the number of adsorbed water molecular layers for some of our samples (0–3 layers) are somewhat consistent with the reported results (5 layers), there is a lack of confidence in our numbers, at this time, due to uncertainties in the surface area data used in this study. More deterministic results may be obtained after the completion of a shale-mudstone surface area study, currently, being conducted; however, this study has clearly demonstrated that samples naturally dried at 4°C have a larger number of water molecular layers adsorbed on their surfaces, compared to those naturally dried at room temperature (25°C). Another point of importance is that this study has shown that most of the adsorbed water is evacuated under vacuum by the time temperatures have reached 50°C, as indicated by the number of layers ( $n_{S50}$ ) at that temperature being 0–1. In previous

studies (e.g. Scromeda and Katsube, 1993, 1994), it was shown that the evacuation of adsorbed or bound water is temperature dependent and that temperatures above 100°C were required for a complete evacuation of adsorbed water, even under vacuum conditions. Further investigation is needed to determine why most of the adsorbed water is evacuated under vacuum with temperatures below 50°C for the samples used in this study. These data, particularly on the number of adsorbed water molecular layers, provide important information that can be used to model the effect of temperature on the permeability history of compacting shale and mudstone under low temperature (<25°C) environments.

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