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Investigation of the Porosity of Certain Iraqi Clay Deposits by Mercury Porosimeter

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Abstract:

Pore volume, pore diameter, and pore volume distribution of three of Iraqi natural clay deposites were measured using mercury intrusion porosimetry .The clays are white kaolin, colored kaolin, and bentonite .The results showed that the variation of the pore area of the clay deposites followed the following order :-

Coloured Kaolin > White Kaolin > Bentonite

While the pore volume may be arranged as in the following sequence:-

White Kaolin > Coloured Kaolin >Bentonite

Also , Bentonite exhibits the narrow range pore size distribution than the white and coloured kaolin.

Key words: Porosity, Iraqi clay, Mercury porosimeter, pore volume, Pore area

Introduction:

Clays are used as industrial raw materials in many applications areas [1,2], including the production of selective adsorbents, bleaching earth, catalyst beds, carbonless copy paper and medication. They are also used as binder in foundries and ceramic production.

The physicochemical properties of these materials play a major role in all these applications .Therefore, the knowledge of structural and porosity properties of such materials is very important. The porous structure can be characterized and measured by various methods, such as, mercury porosimetry, x- ray scattering [3] ,physical adsorption[4] , Electron Atomic Force and Tunnel Microscopy, centrifugal porosotry[5] , displacement of wetting liquids from the pore volume by gas pressure[6] , and others.

Mercury porosimtry can determine a broader pore size distribution more quickly and accurately than other methods. It can be used to characterize pores ranging from 0.003 μm to 360 μm using a single theoretical model.

Mercury intrusion porosimetry and water intrusion porosimetry[7] have been used to explore measurement of the pore structure of filter material containing mixtures of hydrophobic and hydrophilic pores. It has been used to characterize the porosity of two sedimentary limestones built a long the Loire valley in France[8] .A.Jena and K.Gupta[9] measured the pore volume ,pore diameter , pore volume distribution , and pore throat diameter of nano fiber materials using mercury intrusion porosimetry , liquid extrusion porosimetry , and capillary flow porometry .Analysis of result showed that both mercury intrusion and liquid extrusion yielded the same total pore volume and porosity , but pore diameter and distribution were differents.Porosity of certain Iraqi natural silica[10] had been measured using mercury porosimeter.The natural silica are ;glass sand ,standard sand, and flint clay, and the porosity

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parameters measured are pore diameter , pore volume , pore area, and pore size distribution.

In the present work, the porosity of certain Iraqi clay deposits were studied using mercury porosimetry , and the pore volume, pore size , pore surface area , and pore – size distribution were measured too.

Materials and Methods:

The measurements were made using mercury porosimeter , model "Poresizer 9320", obtained from Micromeritics , USA.This can instrument characterize pores ranging from 0.006 μm to 360 μm and capable of generating pressures ranging from 0 to 30.000 psia.

The mercury porosimeter[11,12] is a device which is capable of measuring simultaneously both the pressure and the volume of the mercury taken up by the pores. The measurement was carried out as follows[10]:-

The clay specimen to be examined was weighted usingan analytical balance and dried in vacuum oven at (120 C^o) overnight .After drying process, the specimen was transferred to the low pressure chamber and the measurements proceeded automatically recording the pressure (in psia) and intrusion reading (in pF) (pF=pico farad) .The same procedure was employed after the sample was transferred to the high pressure chamber .The duration – time of the experiment lasted about 5 hours.

Three samples of Iraqi clay deposits have been used .These are Bentonite, White Kaolin, and Coloured Kaolin which have been obtained from State Company of Geological Survey and Mining – Ministry of Industry and Minerals .The specification of these clay are presented[13] in table (1).The samples were ground and sieved and the powder cuts with partical size between (200) and (212) μm were chosed to conduct the measurements.

Table (1) Chemical composition of the Iraqi clay deposits

Sample	%SiO_2	%Al_2O_3	%Fe_2O_3	%TiO_2	%CaO	%MgO	%Na_2O	%K_2O	%L.O.I
White Kaolin	45-64	33.3-38.5	0.9-2.5	0.2-1.7	0.6-1.2	/	/	/	12-14
Coloured Kaolin	45.8-57.1	24.7-29	0.6-9.9	1.3-2.3	0.3	0.3	0.3	/	7.7-13.6
Bentonite	54.6-59.8	13.7-16.7	4.9-5.7	/	2.8-5.8	3.1-3.8	0.7-1.8	0.4-0.9	8.4-13.3

Results and Discussion :

Mercury is non – wetting to most materials because the solid / liquid surface free energy is much higher than the solid / gas surface free energy[14]. Mercury cannot flow into pores spontaneously, but mercury under pressure can be forced into pores. Pressure is used to compute pore diameter. Equating work done due to displacement of mercury at a location in the pore to the increase in surface free energy:-

$$P = -\gamma Cos\theta (dS / dV).......(1)$$

Where P is pressure , γ is the surface tension of mercury , θ is the contact angle of mercury , dV is incremental intrusion volume in the pore and dS is the corresponding incremental solid / liquid surface area. pore diameter (D) is defined as the diameter of a cylindrical opening such that (dS/dV) of the cylindrical opening is equal to the (dS/dV) of that part of pore into which intrusion has occurred .Hence:-

$$D = -4\gamma Cos\ \theta\ /\ p.......(2)$$

The intrusion volume of mercury is the pore volume. Pressure and intrusion volume were measured with better than 0.25% accuracy .The surface tension and contact angle of mercury were 480 dynes/cm and 140^{o} respectively .Table (2a,b,c) shows typical pore size distribution data form and pore area distribution data for (White Kaolin, cloured kaolin and Bentonite respectively).

Calculating the pore diameter intruded by mercury at each pressure requires solving the equation (2). (Washburn equation)[4,9] converting intrusion meter readings to pore volumes requires, first, calculating cumulative changes in capacitance (initial value taken as zero).These changes in capacitance are then multiplied by the conversion factor (pentometer constant) supplied for the penetometer and a units conversion factor to give the cumulative pore volume. Cumulative pore volumes per gram of sample are obtained by dividing by the weight of the sample.

The total pore surface area obtained by assuming that all the pores are cylindrical capillaries .Then the pore surface area (A) for each diameter increment is simply related to incremental pore volume (V) and the average pore diameter (D) by the equation[9]:-

$$A = 4V\ /\ D.......(3)$$

Table (2a) Pore volume and pore area distribution data form for White Kaolin

Pressure Psia	Pore size/ um	Intrusion Reading pF	Cumulative Intrusion pF	Cumulative Por volume cc/gm	Average Pressure psia	Incremental Pore volume cc/gm	Average Pore size um	Incremental Pore area m^2/gm	Cumulative Pore area m^2/gm
0.7	257.14	38.00	------	------	------	-----	-----	------	------
1.1	163.64	37.95	0.05	0.00122	0.9	------	200	-----	0.0000244
1.5	120	37.90	0.1	0.00244	1.3	0.00122	138.462	0.000035	0.000070
2.4	75	37.51	0.49	0.01198	1.95	0.00954	92.308	0.000413	0.00052
3.3	54.55	37.12	0.88	0.0215	2.85	0.00952	63.158	0.000603	0.00136
4.2	42.86	36.80	1.2	0.0293	3.75	0.0078	48	0.00065	0.00244
5.4	33.33	36.46	1.54	0.0376	4.8	0.0083	37.5	0.00089	0.004011
7.1	25.35	36.05	1.95	0.0477	6.25	0.0101	28.8	0.001403	0.00663
8.2	21.95	35.84	2.16	0.0528	7.65	0.0051	23.529	0.00087	0.00898
9.3	19.35	35.60	2.4	0.0587	8.75	0.0059	20.571	0.00115	0.01141
10.3	17.475	35.44	2.56	0.0626	9.8	0.0039	18.367	0.00085	0.0136
11.4	15.789	35.20	2.8	0.0684	10.85	0.0058	16.5899	0.0014	0.0165
12.2	14.75	35.00	3	0.0733	11.8	0.0049	15.254	0.0013	0.01922
12.9	13.95	34.80	3.2	0.0782	12.55	0.0049	14.343	0.00137	0.02181
13.4	13.43	34.65	3.35	0.0819	13.15	0.0037	13.688	0.00108	0.0239
13.6	13.23	34.55	3.45	0.0843	13.5	0.0024	13.333	0.00072	0.0253
13.8	13.04	34.54	3.46	0.0846	13.7	0.0003	13.1387	0.0000913	0.0258
13.8	13.04	34.52	3.48	0.0851	13.8	0.0005	13.0435	0.000153	0.0261
60	3	32.98	3.48	0.0851	----	0	---	---	----
71	2.53	32.90	3.56	0.0870	65.5	0.0019	2.7481	0.00277	0.1266
80	2.25	32.84	3.62	0.0885	75.5	0.0015	1.0596	0.00567	0.3341
100	1.8	32.46	4	0.0978	90	0.0093	2	0.0186	0.1956
186	0.968	29.70	6.76	0.165	143	0.0672	1.2587	0.2136	0.5244
255	0.706	28.10	8.36	0.2044	220.5	0.0394	0.8163	0.1931	1.01593
350	0.514	26.05	10.41	0.2545	302.5	0.0501	0.5950	0.33681	2.1387
511	0.352	24.45	12.01	0.2936	430.5	0.0391	0.4181	0.3741	2.8089
960	0.188	22.40	14.06	0.344	735.5	0.0504	0.2447	0.8239	5.623
1830	0.0984	21.10	15.36	0.3755	1395	0.0315	0.1290	0.977	11.643
2700	0.0667	20.34	16.12	0.3941	2265	0.0186	0.0795	0.9358	19.829
4820	0.0374	19.19	17.27	0.4222	3760	0.0281	0.0479	2.3466	35.257
7474	0.0241	18.49	17.97	0.4393	6147	0.0171	0.0293	2.3345	59.973
9818	0.0183	18.12	18.34	0.4483	8646	0.009	0.02082	1.72911	86.129
10840	0.0166	18.02	18.44	0.4508	10329	0.0025	0.01743	0.5737	103.454
12666	0.0142	17.88	18.58	0.4542	11753	0.0034	0.01532	0.888	118.5901
13838	0.01300	17.82	18.64	0.4557	13252	0.0015	0.0136	0.4412	134.03
14707	0.0122	17.70	18.76	0.4586	14272.5	0.0029	0.01261	0.9199	145.472
15018	0.01198	17.66	18.8	0.4596	14862.5	0.001	0.01211	0.33031	151.808
15050	0.0119	17.66	18.8	0.4596	15034	0	0.01197	0	153.584

Table (2b) Pore volume and pore area distribution data form for cloured Kaolin

Pressure Psia	Pore size/ um	Cumulative Por volume cc/gm	Average Pressure psia	Incremental Pore volume cc/gm	Average Pore size um	Incremental Pore area m^2/gm	Cumulative Pore area m^2/gm
0.8	225	----	---	----	---	---	----
2.5	72	0.0294	1.65	----	109.091	---	0.00108
3.0	60	0.0333	2.75	0.0039	65.455	0.00024	0.00203
4.2	42.86	0.0532	3.6	0.0199	50	0.00159	0.0043
5.3	33.96	0.0622	4.75	0.009	37.895	0.00085	0.0066
6.6	27.27	0.0748	5.95	0.0126	30.252	0.00167	0.0099
7.8	23.08	0.0848	7.2	0.01	25	0.0016	0.0136
8.8	20.45	0.0894	8.3	0.0046	21.687	0.00085	0.0165
9.8	18.37	0.0952	9.3	0.0058	19.355	0.0012	0.01967
10.6	16.98	0.0976	10.2	0.0024	17.6471	0.00054	0.0221
11.9	15.13	0.1044	11.25	0.0068	16	0.0017	0.0261
13.0	13.85	0.1081	12.45	0.0037	14.458	0.00102	0.02991
13.5	13.33	0.1093	13.25	0.0012	13.585	0.00035	0.0322
13.6	13.24	0.1095	13.55	0.0002	13.284	0.0000602	0.03297
13.7	13.14	0.1098	13.65	0.0003	13.187	0.000091	0.0333
66	2.73	0.1098	---	---	---	---	---
73	2.47	0.11098	6935	0.0012	2.58993	0.00185	0.1714
114	1.58	0.117298	93.5	0.00632	1.925	0.0131	0.244
311	0.579	0.16393	212.5	0.0466	0.8471	0.2200	0.7741
436	0.413	0.2025	373.5	0.0386	0.48193	0.3203	1.681
647	0.278	0.2402	541.5	0.0377	0.3325	0.4535	2.8896
1008	0.179	0.284	827.5	0.044	0.2175	0.8092	5.223
1897	0.095	0.3288	1452.5	0.0448	0.1239	1.4463	10.62
2110	0.085	0.3402	2003.5	0.0114	0.08984	0.5076	15.147
3260	0.055	0.3682	2685	0.028	0.06704	1.6706	21.969
4366	0.041	0.3893	3813	0.0211	0.04721	1.7878	32.985
6161	0.029	0.4138	5263.5	0.0245	0.0342	2.865	48.398
8390	0.0215	0.4301	7275.5	0.0163	0.0247	2.6397	69.652
10530	0.0171	0.4396	9460	0.0095	0.01903	1.9968	92.401
11889	0.0151	0.4437	11209.5	0.0041	0.01606	1.0212	110.511
13241	0.0136	0.4473	12565	0.0036	0.01433	1.0049	124.86
14531	0.0124	0.4498	13886	0.0025	0.012963	0.7714	138.795
15300	0.01176	0.4515	14915.5	0.0017	0.012068	0.5635	149.652
16126	0.01116	0.4532	15713	0.0017	0.01146	0.5934	158.185
16414	0.01097	0.4539	16270	0.0007	0.011063	0.2531	164.115
16155	0.01114	0.4544	16284.5	0.0005	0.01105	0.18099	164.489
16010	0.01124	0.455	16082.5	0.0006	0.011192	0.21444	162.62

Table (2c) Pore volume and pore area distribution data form for Bentonite

Pressure Psia	Pore size/ um	Cumulative Por volume cc/gm	Average Pressure psia	Incremental Pore volume cc/gm	Average Pore size um	Incremental Pore area m^2/gm	Cumulative Pore area m^2/gm
0.6	300	----	---	---	---	---	---
1.2	150	0.0027	0.9	----	200	---	0.000054
1.6	112.5	0.0053	1.4	0.0026	128.571	0.000081	0.000165
2.2	81.82	0.0102	1.9	0.0049	94.737	0.00021	0.000431
3.0	60	0.0271	2.6	0.0169	69.231	0.00098	0.00157
4.1	43.90	0.0857	3.55	0.0586	50.704	0.00462	0.0068
4.6	39.13	0.125	4.35	0.0393	41.379	0.0038	0.0121
5.2	34.62	0.164	4.9	0.039	36.735	0.0042	0.018
6.1	29.51	0.2199	5.65	0.0559	31.858	0.0070	0.028
7.0	25.71	0.2599	6.55	0.04	27.481	0.0058	0.0378
9.2	19.57	0.296	8.1	0.0361	22.222	0.0065	0.0533
10.0	18	0.301	9.6	0.005	18.75	0.0011	0.0642
11.2	16.07	0.3141	10.6	0.0131	16.9811	0.0031	0.07399
12.0	15	0.319	11.6	0.0049	15.517	0.00124	0.0822
12.8	14.06	0.322	12.4	0.003	14.5161	0.00083	0.0887
13.1	13.74	0.323	12.95	0.001	13.8996	0.00029	0.09295
13.5	13.33	0.326	13.3	0.003	13.5338	0.00089	0.0964
13.7	13.139	0.327	13.6	0.001	13.235	0.00030	0.09883
13.8	13.04	0.3272	13.75	0.0002	13.091	0.000061	0.09998
60	3	0.3272	---	---	---	---	---
75	2.4	0.3323	67.5	0.0051	2.67	0.0076	0.498
80	2.25	0.3341	77.5	0.0018	2.323	0.0031	0.5753
90	2	0.33372	85	0.0031	2.1176	0.0059	0.6369
100	1.8	0.3398	95	0.0026	1.895	0.0055	0.7173
961	0.187	0.3682	530.5	0.0284	0.3393	0.283	4.341
2240	0.080	0.3765	1600.5	0.0083	0.11246	0.2952	13.359
4540	0.0396	0.384	3390	0.0075	0.0531	0.565	28.927
7133	0.0252	0.3931	5836.5	0.0091	0.0308	1.182	51.052
9183	0.0196	0.3999	8158	0.0068	0.0221	1.231	72.380
12841	0.0140	0.4089	11012	0.009	0.01635	2.202	100.037
13719	0.0131	0.4095	13280	0.0006	0.0136	0.1765	120.441
14230	0.01265	0.4122	13974.5	0.0027	0.0129	0.8372	127.814
15151	0.0119	0.4146	14690.5	0.0024	0.01225	0.784	135.379
15518	0.01159	0.41597	15334.5	0.00137	0.01174	0.4668	141.73
15540	0.01158	0.4169	15529	0.00093	0.01159	0.321	143.883

The cumulative surface area for each point is the sum of these for all preceding points .Table (3) summarized the experimental values of pore volume, pore area, and medium pore diameter on the three different Iraqi natural clays.

Table (3) The porosity parameters of the Iraqi clay deposits

Sample	**Pore volume cc/gm**	**Pore Area m^2/gm**	**Mediam pore diameter** μm
White Kaolin	0.4596	153.58	0.0126
Coloured Kaolin	0.4550	162.62	0.0112
Bentonite	0.4169	143.88	0.0129

The value of D on the distribution curve corresponding to the maximum value of $\Delta V / \Delta D$ is termed the median pore diameter and also called the most abundant pore diameter.

he results indicate that the pore area of the three clay deposides varied in an order that may be arranged in sequence as:-

Coloured Kaolin > White Kaolin > Bentonite

While the pore volume of that deposides varied in an order and can be arranged as follows:-

White Kaolin > Coloured Kaolin > Bentonite

The pore area of cloured kaolin is larger than that of white kaolin and bentonite , this may be because the coloured kaolin has a some metals can be analyzed in the crystal structure .The pore volume of white kaolin is larger than of coloured kaolin , which is agreement with the results obtained on pore diameters.

The differential pore size distributions were estimated from the plot $\Delta V / \Delta D$ against D as tabulated in Table (4) and indicated in figs. (1-3)

According to the IUPAC classification one can distinguish three different type of pore :-micro-(< 2 nm) ,meso-(2-50 nm) , and macropores (> 50 nm)[4] .

The analysis of result obtained using this technique showed that the total pore volume associated with the mesopore accounted for (0.0655), (0.0868), and (0.0404) of the total volume for (White Kaolin), (Coloured Kaolin), and (Bentonite) respectively.

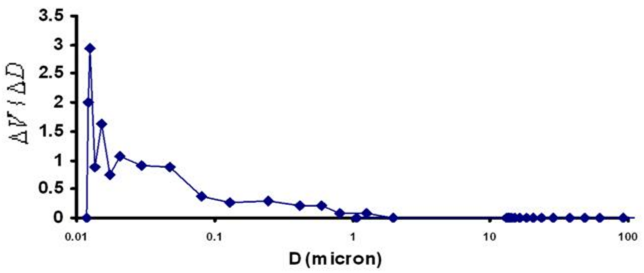
Also, the pore volumes for (0.3941), (0.3682) ,and (0.3765) associated with macropores accounted for (White Kaolin) ,(Coloured Kaolin) ,and (Bentonite) respectively.

The results also indicate that the Bentonite exhibits the narrow range pore size distribution than the white and coloured kaolin.

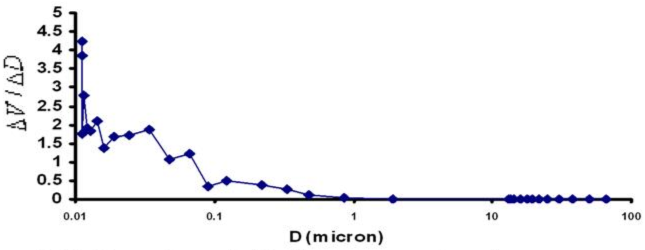
Table (4) the data of pore size distributions for the three types of clay

White Kaolin		**Coloured Kaolin**		**Bentonite**	
$\Delta V / \Delta D$	**D**	$\Delta V / \Delta D$	**D**	$\Delta V / \Delta D$	**D**
0.000198	138.462	0.0000893	65.455	0.000036	128.571
0.00021	92.308	0.001288	50	0.000145	94.737
0.00033	63.158	0.00743	37.895	0.00066	69.231
0.000515	48	0.00165	30.252	0.0032	50.704
0.00079	37.5	0.00190	25	0.0042	41.379
0.00116	28.8	0.00139	21.687	0.0084	36.735
0.00097	23.529	0.00249	19.355	0.0115	31.858
0.00199	20.571	0.00141	17.6471	0.00914	27.481
0.00177	18.367	0.00413	16	0.0069	22.222
0.0033	16.5899	0.002399	14.458	0.00144	18.75
0.00367	15.254	0.001375	13.585	0.00741	16.9811
0.0056	14.343	0.00066	13.284	0.00335	15.517
0.00565	13.688	0.00310	13.187	0.002997	14.5161
0.0068	13.333	0.009504	1.925	0.00162	13.8996
0.00154	13.1387	0.04323	0.8471	0.0082	13.5338
0.0053	13.0435	0.1057	0.48193	0.0033	13.235
0.00089	1.0596	0.253	0.3325	0.00139	13.091
0.0099	2	0.383	0.2175	0.0052	2.323
0.091	1.2587	0.4786	0.1239	0.0151	2.1176
0.0891	0.8163	0.3343	0.08984	0.0117	1.895
0.227	0.595	1.2281	0.06704	0.0183	0.3393

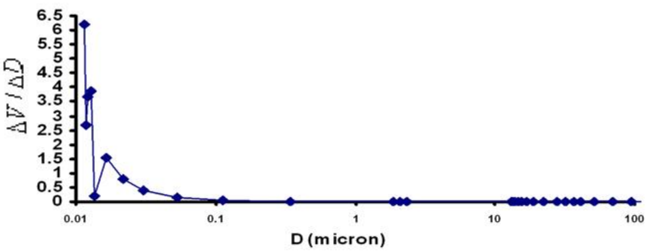
0.221	0.4181	1.0657	0.04721	0.0366	0.11246
0.291	0.2447	1.885	0.0342	0.1263	0.0531
0.2723	0.129	1.7158	0.0247	0.4081	0.0308
0.376	0.0795	1.667	0.01903	0.782	0.0221
0.8892	0.0479	1.3805	0.01606	1.552	0.01635
0.9194	0.0293	2.1176	0.01433	0.2143	0.0136
1.059	0.02082	1.8248	0.012963	3.857	0.0129
0.7375	0.01743	1.8994	0.012068	3.6923	0.01225
1.6190	0.01532	2.7869	0.01146	2.6863	0.01174
0.872	0.0136	1.7632	0.011063	6.2	0.01159
2.9293	0.01261	3.8462	0.01105		
2	0.01211	4.225	0.011192		
0	0.01197				



Fig(1) :Pore volume distribution over pore diameter for White Kaolin



Fig(2) :Pore volume distribution over pore diameter for Coloured Kaolin



Fig(3) :Pore volume distribution over pore diameter for Bentonite

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التحري عن مسامية بعض ترسبات الاطيان العراقية باستخدام مقياس المسامية الزئبقي

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الخلاصة:

استخدم مقياس المسامية الزئبقي (Mercury Porosimeter) لقياس كل من حجم المسام وقطر المسام وتوزيع حجم المسام ،لثلاثة انواع من خامات الاطيان العراقية ،الكاؤولين الابيض والكاؤولين الملون والبنتونايت.واظهرت نتائج القياسات ان مساحة المسام لهذه الاطيان تتبع التسلسل الاتي:-

الكاؤولين الملون > الكاؤولين الابيض > البنتونايت

بينما كان حجم المسام يتغير وفق الترتيب:-

الكاؤولين الابيض > الكاؤولين الملون > البنتونايت

ووجد كذلك ان اقطار مسام البنتونايت تتوزع بمدى اضيق من كل من الكاؤولين الابيض والكاؤولين الملون.

كلمات مفتاحية: المسامية، اطيان عراقية، مقياس المساميةالزئبقي، حجم المسام، مسافة المسام