Lamp : 1. Karakterisasi Bentonit Alam Menggunakan XRD

Diffractometer type: PW 3710 BASED
Tube Anode: Cu
Generator tension [kV] : 40
Generator current [mA] : 30
Wavelength Alpha 1 [Å] : 1.54056
Wavelength Alpha 2 [Å] : 1.54439

Angle [2θ]	d-value α 1 [Å]	d-value α 2 [Å]	Peak width [2θ]	Peak int [counts]	Back int [counts]	Rel. int [%]	Sigif
12.175	7.2636	7.2816	0.400	34	67	5.70	1.96
19.720	4.4982	4.5095	0.320	52	81	8.8	1.43
20.710	4.2854	4.2960	0.080	166	88	28.2	1.24
24.935	3.568	3.5769	0.640	17	92	2.8	1.2
26.480	3.3632	3.3716	0.120	590	92	100	6.39
27.800	3.2065	3.2144	0.320	32	92	5.5	0.81
31.740	2.8168	2.8238	0.480	14	79	2.3	1.01
34.835	2.5733	2.5797	0.240	48	79	8.1	1.42
36.385	2.4672	2.4733	0.120	50	77	8.5	1.93
39.300	2.2906	2.2963	0.120	46	76	7.8	2.04
42.290	2.1353	2.1406	0.240	32	74	5.5	1.97
45.450	1.994	1.9989	0.640	6	76	1	0.95
49.965	1.8238	1.8284	0.080	69	71	11.7	1.4
54.665	1.6776	1.6818	0.160	24	76	4.1	0.91
59.775	1.5458	1.5497	0.120	37	72	6.3	0.94
61.490	1.5068	1.5105	0.800	15	72	2.6	1.25
67.985	1.3778	1.3812	0.120	28	71	4.8	0.94

ASTM

13-135 MAJOR CORRECTION

d	15.0	4.50	5.01	15.0
I/I$_1$	100	80	60	100

$Ca_{0.24}Na_{0.01}Mg_{0.36}Fe_{0.02}Al_{1.75}Si_{3.87}O_{10}(OH)_2 \cdot 1.078H_2O$

CALCIUM MAGNESIUM ALUMINUM SILICATE HYDRATE — MONTMORILLONITE

Rad. λ Filter Dia.
Cut off I/I$_1$, DIFFRACTOMETER
Ref. ROSENQUIST, NORSK GEOL. TIDSSKR. 39 350 (1959)

Sys. S.G.
a_o 5.2 b_o 9.0 c_o 15.0 A C
α β γ Z Dx
Ref.

ε α nωβ εγ Sign
2V D mp Color
Ref.

SAMPLE FROM SKYRVEDALEN, HEMSEDAL, NORWAY.
ANALYSIS %: 59.58 SiO_2, 22.96 Al_2O_3, 0.47 Fe_2O_3, 3.67 MgO, 3.38 CaO, 0.06 Na_2O, LOSS 110°–950° 9.61.
*EXPANDS TO 18.0 WITH GLYCEROL TREATMENT.

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
15.0*	100	001			
5.01	60	002			
4.50	80	110,020			
3.77	20	004			
3.50	10				
3.30	10				
3.02	60	005			
{2.55	40	200			
{2.50		006			
2.26	10				
2.1[illegible]	10	007			
1.88	10	008			
1.70	30	009			
1.50	50	060			
1.493	50				
1.285	20				
1.243	20				

16-0221 MAJOR CORRECTION

d	3.58	7.18	1.49	7.18
I/I$_1$	100+	100	100	100

$Al_2Si_2O_5(OH)_4$

ALUMINUM SILICATE HYDRATE — KAOLINITE (B-AXIS DISORDERED FORM)

Rad. CoKα λ 1.7902A Filter Fe
Dia. 19,20cm Cut off Coll.
I/I$_1$ VISUAL d corr. abs.?
Ref. ROBERTSON, BRINDLEY, MACKENZIE, AM. MIN. 39 118-38 (1954)

Sys. PSEUDO-MONOCLINIC S.G.
a_o 5.16 b_o 8.93 c_o 7.39 A 0.578 C 0.828
α β 104.5° γ Z 2 Dx 2.600
Ref. IBID.

ε α nωβ εγ Sign
2V D mp Color WHITE
Ref. IBID.

KAOLIN CLAY FROM PUGU, TANGANYIKA. ALSO ELECTRON, OPTICAL AND DIFFERENTIAL THERMAL DATA AND CHEMICAL ANALYSIS GIVEN.

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
7.18	100	001	1.310	10s	204
4.48	80s	02-	1.287	30	261,40$\bar{1}$
3.58	100+	002			{33$\bar{1}$,062
2.565	80	20$\bar{1}$,130	1.265	10	{332
2.502	80	13$\bar{1}$,200	1.249	5	26$\bar{2}$,400
2.386	80	003	1.236	30s	40$\bar{3}$,261
2.341	90s	20$\bar{2}$,131	1.194	10	006
2.206	10s	132,201			
1.989	40s	20$\bar{3}$,132			
1.789	40	004			
1.666	50s	20$\bar{4}$,133			
1.541	10s	13$\bar{4}$,203			
1.488	100	060,33$\bar{1}$			
1.458	30s	{33$\bar{2}$,061			
		{330			
1.432	20	005			
1.375	10s	{33$\bar{3}$,062			
		{331			
1.339	10	13$\bar{5}$			

5-0490 MINOR CORRECTION

d	3.34	4.26	1.82	4.26
I/I$_1$	100'	35	17	35

SiO_2

SILICON IV OXIDE — ALPHA QUARTZ

Rad. $CuK\alpha_1$ λ 1.5405 Filter Ni
Dia. Cut off Coll.
I/I$_1$ G.C. DIFFRACTOMETER d corr. abs.?
Ref. SWANSON AND FUYAT, NBS CIRCULAR 539, VOL. III (1953)

Sys. HEXAGONAL S.G. D_3^4 – P3$_1$21
a_o 4.913 b_o c_o 5.405 A C 1.10
α β γ Z 3
Ref. IBID.

ε α nωβ 1.544 εγ 1.553 Sign +
2V D_x 2.647 mp Color
Ref. IBID.

MINERAL FROM LAKE TOXAWAY, N.C. SPECT. ANAL.: <0.01% Al; <0.001% Ca, Cu, Fe, Mg.
X-RAY PATTERN AT 25°C.

REPLACES 1-0649. 2-0458, 2-0459, 2-0471, 3-0419, 3-0427, 3-0444

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
4.26	35	100	1.228	2	220
3.343	100	101	1.1997	5	213
2.458	12	110	1.1973	2	221
2.282	12	102	1.1838	4	114
2.237	6	111	1.1802	4	310
2.128	9	200	1.1530	2	311
1.980	6	201	1.1408	<1	204
1.817	17	112	1.1144	<1	303
1.801	<1	003	1.0816	4	312
1.672	7	202	1.0636	1	400
1.659	3	103	1.0477	2	105
1.608	<1	210	1.0437	2	401
1.541	15	211	1.0346	2	214
1.453	3	113	1.0149	2	223
1.418	<1	300	0.9896	2	402,115
1.382	7	212	.9872	2	313
1.375	11	203	.9781	<1	204
1.372	9	301	.9762	1	320
1.288	3	104	.9607	2	321
1.256	4	302	.9285	<1	410

Lamp: 2. Penentuan Komposisi Bentonit Dengan AAS

2.1. Bentonit Alam Dan Bentonit Natrium

Berat sampel kering = 200 g

Volume sampel saat analisa = 100mL

Faktor pengenceran, fp = 5

Faktor kimia, fk =BM. / BA

$$\% \text{ Berat Oksida} = \frac{C.fp\ mg\ V\ mL.fk.100}{1000\,mL\ 200\,mg}\ \%$$

Bentonit Alam				
Logam	Abs	Kurva Standar	Konsentrasi (ppm)	% Berat Oksida
Aluminium	0,103	y = 0,0025 x	41,2	19,46
Magnesium	0,438	y = 0,0645 x	6,79	1,70
Kalsium	0,133	y = 0,0195 x	6,82	1,71
Natrium(*)	0,183	y = 0,1300 x	1,41	1,41
Bentonit Natrium				
Logam	Abs	Kurva Standar	Konsentrasi (ppm)	% Berat Oksida
Aluminium	0,109	y = 0,0025 x	46,6	20,60
Magnesium	0,426	y = 0,0645 x	6,60	1,65
Kalsium	0,052	y = 0,0195 x	2,67	0,67
Natrium(*)	0,205	y = 0,1300 x	1,58	1,58

(*) fp=20

2.2.Bentonit Alam dan Bentonit Kalsium

Jenis Bentonit	Oksida	% Berat
Alam	CaO	1,13
Kalsium	Al_2O_3	17,95
	MgO	1,62
	CaO	1,49
	Na_2O	0,61

Lamp: 3. Penghitungan Kapasitas Berdasarkan Komposisi Bentonit Natrium

3.1 Perhitungan Kapasitas

$$\text{mek} = \frac{C.fp.mg100mL.1000mg.valensi}{1000mL.200mg.BAmg.mmol^{-1}}$$

Kation	Konsentrasi	Kapasitas (mek)
Ca	2,67	$\frac{2,67.5.100.5.2}{1000.40.08} = 0,333$
Na	1,58	$\frac{1,58.20.100.5.2}{1000.22,99} = 0,6973$
Mg	6,60	$\frac{6,60.5.100.2}{1000.24,31} = 1,3714$
	Jumlah	2,3714

3.2 Preparasi Larutan $CdSO_4$ 8/3 H_2O 0,02N (100 mL . 0,02 N= 2mek)

- Larutan $CdSO_4$ 8/3 H_2O 0,04 M = 0,08 N, 500 mL

  BM $CdSO_4$ 8/3 H_2O = 256,5 $g.mol^{-1}$ (lang's Hand Book)

  0,04 M = x.1000/256,5.500

  x = 5,128 g

- Larutan $CdSO_4$ 8/3 H_2O 0,01 M= Cd^{2+} 0,01M

  BA Cd = 112,4 $g.mol^{-1}$

  0,01 M = 0,01 $mol.L^{-1}$.112,4 $g.mol^{-1}$= 1,124 $g.L^{-1}$

  = 1124 ppm

Lamp: 4 Penentuan Bilangan Hellferich

$$He \cong \frac{\overline{C}\overline{D}\delta}{CDr}(5+2\alpha_{AB})$$

Jika He « 1 , kecepatan pertukaran ditentukan oleh difusi intrapartikel
He » 1, kecepatan pertukaran ditentukan oleh difusi ion melalui film

Bila $\overline{C}$ = 2 mek/g,
V bentonit (basah) = 6,3 mL
maka $\overline{C}$ = 0,3175 mg/mL
d = 200# = 16.000 µm /200
= 80 µm = 800.000 Å

r = 400.000 Å
α_{AB} = tebal air serupa kristal
Bentonit Na δ = 7,5 Å
Ca δ = 10 Å
Max δ = 60 A

C = 713,44 ppm = 713,44 (mg/mL).(ek/ 56,2.1000mg)
= 0,0127 N = 0,0127 mek/mL

Bentonit Na, δ = 7,5 Å

$$He \cong \frac{0,3175(5+2)7,5}{0,0127.400.000}\frac{\overline{D}}{D}$$

$$\cong 0,0031\frac{\overline{D}}{D}$$

$$\frac{\overline{D}}{D} < 1 \Rightarrow He \ll 1$$

Bentonit Ca, δ = 10 Å

$$He \cong \frac{.0,3175(5+2)10}{2.\ 0,0127.400.000}\quad \frac{\overline{D}}{D} = 0,0022\frac{\overline{D}}{D}$$

$$\frac{\overline{D}}{D} < 1 \Rightarrow He \ll 1$$

Max, δ Bentonit = 60 Å

$$He \cong \frac{0,3175.60.(5+2)}{0,0127.400.000}\frac{\overline{D}}{D} = 0,0263\frac{\overline{D}}{D}$$

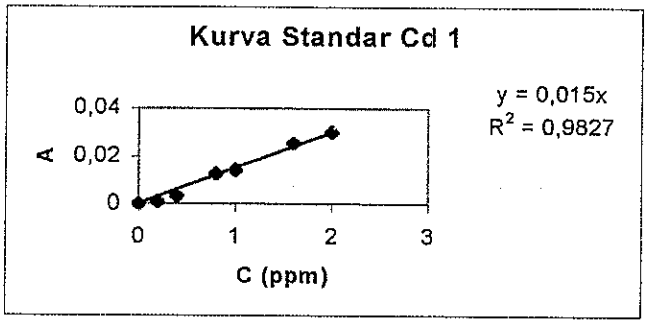
$$\frac{\overline{D}}{D} < 1 \Rightarrow He \ll 1$$

Lamp: 5. Pengambilan Kadmium Menggunakan Penukar Ion - Bentonit

5.1 Pengambilan Menggunakan Bentonit Alam Dan Bentonit Natrium

Larutan Standar Cd 1

C (ppm)	Abs
0	0
0,2	0,001
0,4	0,0032
0,8	0,0125
1	0,0140
1,6	0,0252
2	0,0299



Bentonit Alam

Kode Sampel	Lama Perlakuan (jam)	Abs	Konsentrasi Kadmium Sisa		Tertukarkan (ppm)
			Teramati (ppm)	Total (ppm)	
0	0	0,0182	1,12	713,44	
A1	1	0,0130	0,8667	509,6	203,84
A2	2	0,0130	0,8667	509,6	203,84
A3	3	0,0159	1,06	623,28	89,56
A6	6	0,0177	1,18	693,84	19,6
A12	12	0,0137	0,9133	537,04	176,4
A24	24	0,0145	0,9667	568,4	145,04
A48	48	0,0061	0,4067	293,12	474,32

Bentonit Natrium

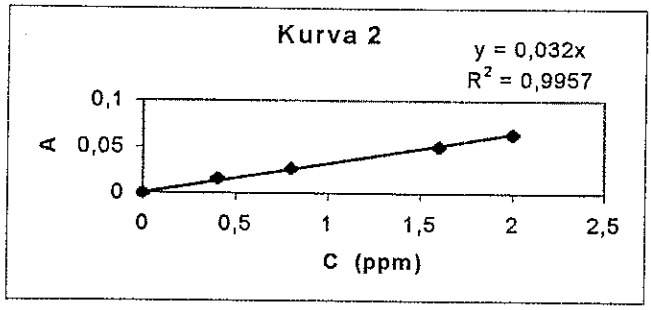
Kode Sampel	Lama Perlakuan (jam)	Abs	Konsentrasi Kadmium Sisa		Tertukarkan (ppm)
			Teramati (ppm)	Total (ppm)	
Na1,5	1,5	0,0122	0,8133	478,24	234,2
Na3	3	0,0139	0,9261	544,88	168,56
Na6	6	0,0147	0,98	576,24	137,2
Na12	12	0,0148	0,9867	580,16	133,28
Na24	24	0,0098	0,6533	384,16	329,28
Na48	48	0,0078	0,52	305,76	407,68

Nb: faktor pengenceran= fp =588

5.2 Pengambilan Oleh Bentonit Natrium Dan Bentonit Kalsium

- Larutan Standar Cd 2

C (ppm)	Abs
0	0
0,4	0,0157
0,8	0,0269
1,6	0,0507
2	0,0638

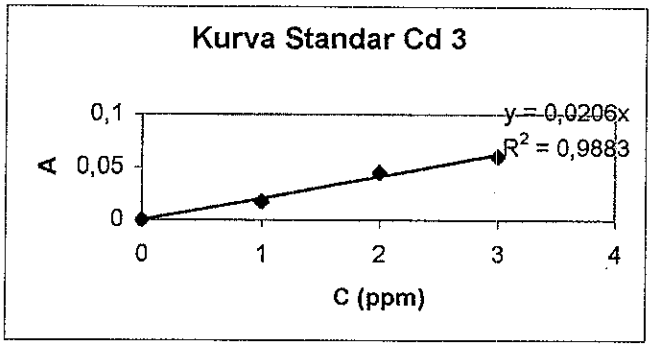


Kode Sampel	Lama Perlakuan (jam)	Abs	Konsentrasi Kadmium Sisa	
			Teramati (ppm)	Total (ppm)
	Bentonit Natrium			
Na1000p°		0,3571	11,121	111,21*
Na750p	1,5	0,0360/2	0,5607	329,72
Na500p	1,5	0,0418/4	0,3255	192,42
Na250p	1,5	0,0140/8	0,0545	32,05
Na125p	1,5	0,0077/8	0,0300	17,64
	Bentonit Kalsium			
Ca1,5	1,5	0,0067	0,2094(1)	
Ca6	6	0,0703	2,1969	
Ca12	12	0,0685	2,1406	
Ca24	24	0,0662	2,0688	
Ca48	48	0,0651	2,0344	

Faktor pengenceran = fp= 588
*fp = 10

Larutan Standar Cd 3

C (ppm)	Abs
0	0
1	0,0178
2	0,0450
3	0,0600



Kode Sampel	Lama Perlakuan (jam)	Abs	Konsentrasi Kadmium Sisa (ppm)	
	Larutan Awal		Teramati	Total
0		0,209	1,0146	596,56
1000p°		0,0611/2,5	1,1864	593,20**
	Bentonit Kalsium			
Ca1,5	1,5	0,0094	0,4563(2)	268,30
Ca3	3	0,0182	0,0180	519,50
Ca6	6	0,0436	2,1165	1244,50
Ca12	12	0,0425	2,0631	1213,10
Ca24	24	0,0458	2,2233	1307,34
Ca48	48	0,0440	2,1359	1255,90

Ca1,5 = (1) + (2) / 2
Faktor pengenceran = fp = 588
**fp = 500

Ho : $\mu 1 = \mu 2$
$\alpha = 0,05$

	Konsentrasi Cd teramati		di	di - $\overline{X_D}$
	Menurut Kurva std 2	Menurut Kurva std 3		
Ca6	2,1969	2,1165	0,0804	0,011004
Ca12	2,1406	2,0631	0,0775	0,010404
Ca 24	2,0688	2,2233	-0,1545	0,016900
Ca48	2,0344	2,1359	-0,1015	0,005929
			$\overline{X_D}$ -0,0245	Σ0,044237

$$S_D = \sqrt{\frac{1\sum\left(di - \overline{x_D}\right)^2}{n-1}} = \sqrt{\frac{1\sum\left(0,044237\right)}{4-1}} = 0,1214$$

$$thit = \frac{x_D - 0}{S_D/\sqrt{n}} = \frac{-0,0245}{0,1214/\sqrt{4}} =$$

t tabel = $t_{3,\ 0,025}$ = - 3,182

t hitung > t tabel , maka Ho diterima.

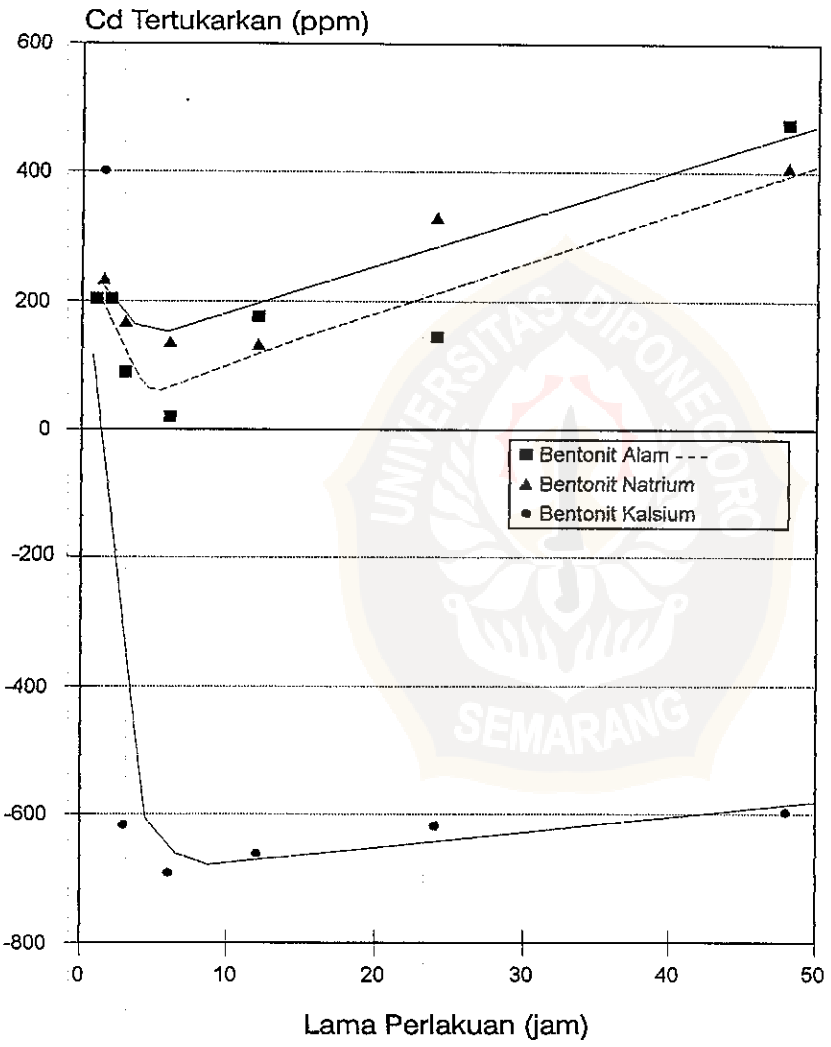
Pengambilan Oleh Bentonit Natrium Selama 1,5 jam

Kode Sampel	Konsentrasi Cd Awal C° (ppm)	Cd Tertukarkan Ct (ppm)	Ct / C°(%)
Na1,5	713,44	234,2	33
Na750p	444,90	114,15	26
Na500p	296,60	104,56	35
Na250p	148,30	116,14	78
Na125p	74,15	56,51	76

Pengambilan oleh Bentonit Kalsium

Lama Perlakuan (jam)	Cd Tertukarkan (ppm)
1,5	400,81
3	-617,61
6	-691,52
12	-661,52
24	-619,43
48	-598,26

Grafik Pengambilan Cd oleh Penukar-Ion Bentonit Selama 1,5-48 Jam



Lamp: 6. Penentuan Kapasitas Pertukaran Bentonit Natrium Selama 1,5 Jam Menggunakan Isoterm Langmuir [25]

$$1/q_e = \frac{1}{bq^\circ}\ 1/C_e + 1/q^\circ$$

$$y = m\ x + C$$

q° = kapasitas pertukaran
C_e = konsentrasi Cd sisa
q_e = konsentrasi Cd tertukarkan

$$\text{ppm} = \frac{\text{mg}}{1000\text{mL}.\text{BM}(\text{mg}/\text{mmol})} = \text{M}$$

C_e		q_e		$1/C_e.10^-$	$1/q_e.10^4$	xy	X^2
ppm	r.10^4 M	ppm	s.10^4 M	1/r =x	1/s =y		
17,69	1,5738	56,46	5,0231	0,6354	0,1991	0,1265	0,4037
32,16	2,8612	116145	10,3327	0,3495	0,0968	0,0338	0,1222
192,04	17,0854	104,56	9,3025	0,0585	0,1075	0,0063	0,0034
330,75	29,4262	114,15	10,1557	0,340	0,0985	0,0033	0,0012
478,24	42,5480	234,2	20,8363	0,0235	0,0480	0,0011	0,0006
		Jumlah		1,1009	0,5499	0,1710	0,5311

$$M = \frac{n\sum xy - \sum x.\sum y}{n\sum x^2 - \left(\sum x\right)^2} =$$

$$= \frac{5.0,1710 - 1,1009.0,5499}{5.0,5311 - (1,1009)^2} = 0,1729$$

$$m = \overline{y} - m\,\overline{x}$$

$=0,5499/5-0,01729.1,1009/5= 0,0719= 1/q^\circ=13,9082$

$q^\circ = 13,9082(10^{-4})$ M = 156,33 ppm = 0,28 mek

$b= 1/mq^\circ \Rightarrow K = mq^\circ = 0,1722.13,9276.10^{-4}$

$= 2,4 .10^{-4}$

Lamp: 7. Analisa Pengaruh Konsentrasi Terhadap Pertukaran

Asumsi : counter ion homogen/tunggal

Diketahui : $Kc = 2{,}4 \cdot 10^{-4}$

Volume bentonit (basah) = 6,3 mL

Pertukaran Ion Valensi Sama: 2+

$BCa^{2+} + Cd^{2+} \rightleftharpoons BCd^{2+} + Ca^{2+}$

Pers:

$$\frac{\bar{x}_A}{(1-\bar{x}_A)} = Kc.\frac{x_A}{(1-x_A)}$$

Pertukaran Ion Valensi Berbeda

$2BNa^{+} + Cd^{2+} \rightleftharpoons B2Cd^{2+} + 2Na^{+}$

Pers:

$$\frac{\bar{x}_C}{(1-\bar{X}_c)^2} Kc \frac{[C^{2+}]x_C}{[C^{2+}](1-x_C)^2}$$

7.1 Pertukaran Antara Ca^{2+} Dengan Cd^{2+}

Bentonit: $\bar{c}$ = 2mek =1mmol = a.10-4 M.6,3 mL, a = $1{,}5873.10^3$

$\bar{x}$	$1-\bar{x}$	$\bar{x}/(1-\bar{x})$
$5{,}0231 / 1{,}5873.10^3 = 0{,}0032$	0,9968	0,0032
$10{,}3327/ 1{,}5873.10^3 = 0{,}0065$	0,9935	0,0065
$9{,}3025/ 1{,}5873.10^3 = 0{,}0059$	0,9941	0,0059
$10{,}1557/ 1{,}5873.10^3 = 0{,}0064$	0,9936	0,0064
$20{,}8363/ 1{,}5873.10^3 = 0{,}0132$	0,9868	0,0134

Larutan C^o=713,44ppm= 713,44mg/1000mL.112,4mg.$mmol^{-1}$=$63{,}47.10^{-4}$M

x	1-x	x/1-x
1,5738 / 63,47=0,0248	0,9752	0,0254
2,8612/ 63,47=0,0451	0,9549	0,0472
17,0854/ 63,47=0,2692	0,7708	0,3492
29,4262/ 63,47=0,4636	0,5364	0,8643
42,5480/ 63,47=0,6704	0,3296	2,0340

7.2 Pertukaran Antara Na^+ Dengan Cd^{2+}

Bentonit: $\overline{C}$ = 2mek= 2mmol = a.10-^{4}.6,3 mL, a = 3,1746.10^3

$\overline{C}' = \overline{\overline{C}} - q_e$

$\bar{x}$	$(1-\bar{x})^2$	$\bar{x}/(1-\bar{x})^2$
5,0231 / 3,1696.10^{-3}=0,0016	0,9968	0,0016
10,3327 / 3,1643.10^{-3}=0,0033	0,9934	0,0033
9,3025 / 3,1653.10^{-3}=0,0029	0,9942	0,0029
10,1557 / 3,1644.10^{-3}=0,0032	0,9936	0,0032
20,8363 / 3,1538.10^{-3}=0,0066	0,9868	0,0067

$C^{o'} = C_e + 2q_e = C^o + q_e$

Larutan C^o=713,44= 713,44mg.mmol^{-1}/1000mL.112,4mg.mmol^{-1}=63,47.10^{-4}M

x	$(1-x)^2$	$x/(1-x)^2$
17,69 / 68,4976 = 0,0230	0,9545	0,0241
32,16 / 73,8128 = 0,0388	0,9239	0,0420
192,04 / 72,7173 = 0,2346	0,5858	0,4005
330,75 / 73,71773= 0,3992	0,3610	1,1058
478,24 / 84,3063 = 0,5047	0,2453	2,0575

Pertukaran antar ion Divalen	Pertukaran Antara Ion monovalen dan Ion Divalen
$\frac{\bar{x}_A}{(1-\bar{x}_A)} = Kc.\frac{x_A}{(1-x_A)}$	$\frac{\bar{x}_C}{(1-\bar{x}_c)^2} Kc \frac{[C^{2+}]x_C}{[C^{2+}](1-x_C)^2}$
0,0032 ≅ 0,0601.10^{-4}	0,0016 ≅ 2,6763.10^{-4}
0,0065 ≅ 0,1133.10^{-4}	0,0033 ≅ 4,3213.10^{-4}
0,0059 ≅ 0,8381.10^{-4}	0,0029 ≅ 43,3543.10^{-4}
0,0065 ≅ 2,0743.10^{-4}	0,0032 ≅ 113,9328.10^{-4}
0,0133 ≅ 4,8816.10^{-4}	0,0067 ≅ 184,7306.10^{-4}