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สถาบันวิทยบริการ
จุฬาลงกรณ์มหาวิทยาลัย

APPENDICES

APPENDIX A

Slip preparation for deflocculant test and flow measurement

Apparatus

1. Motor mixer, Heidolph RZR 2041
2. Beaker 600 ml.
3. Brookfield viscometer model LVT spindle No.2, 60 rpm.

Procedure

1. Calculate and weigh 1000 g. of desired sample (by dry weight).
2. Add DI water (in the proportion by dry weight of clay) and allow to slake for a while 15 min approximately.
3. Mix with motor mixer at speed of 650-700 rpm., add sodium silicate if necessary to obtain the flow, pass through 200 mesh.
4. Transfer to the container and measure slip density, solid content and amount of used sodium silicate.
5. Pass the slip to the magnet separator 2-3 times to eliminate ferromagnet in the slip. Age for 24 hr. before use.

Deflocculant test

1. Use 600 ml. of prepared slip (known solid content) to stir and gradually add Na_2SiO_3 (if the binder used, add the binder before adding sodium silicate), stir 2 min for each step.

2. Pour in the 600 ml. beaker at 500 ml. level, measure the viscosity after rotating spindle 10 rounds.
3. Record the viscosity until flocculation occur or unchangeable ready.
4. Calculate the deflocculant use in each step and plot curve between % Na_2SiO_3 and viscosity.

**Shear measurement by Brookfield small sample DVII+
(Degree of shear thinning and thixotropy)**

1. Use one litre of slip already mixed with additives and stir with mixer for 2 min at 1000 rpm. (measure solid content)
 2. Set rpm. for test, start at 0.3, 0.6, 1.5, 3.0, 6.0, 12.0, 30.0 and 60.0 rpm. and select spindle No. (No. 18 for lower viscosity, No. 31 for higher viscosity)
 3. Pour in the sample container at the half level screw spindle on the shaft level and put in the sample.
 4. Turn on the motor and start at 0.3 rpm. for 2 min, record cP (viscosity), SS (shear stress), SR (Shear rate)
 5. Change to higher speed without turning off the motor and record each step until to 60 rpm.
 6. Decrease the speed in steps to the slowest speed without turning off the motor and record each step.
 7. Stop 5 min then turn on at 0.3 rpm. for 2 min and record the value.
- (Only shear response in increasing shear rate step for rechecking.)

Slip preparation for Modulus of rupture

Apparatus

1. Plaster
2. Motor mixer

Procedure

1. Use the prepared slip for deflocculation 600 ml. at 50% solid.
2. Add Na_2SiO_3 in the amount of testing.
3. Mix for 10 min at speed 1050 rpm., screen through 40 mesh.
4. Cast in cylindrical plaster mould and dry at 110°C for 24 hr.
5. Keep in desiccator before breaking with 3-point bending tester (Hungta Instrument).
6. Calculate the MOR from equation (4.4), use 8-test pieces for average value.

APPENDIX B

JCPDS cards and diffraction data

Kaolinite 14-0164

14-0164		Wavelength= 1.5418									
Al ₂ Si ₂ O ₅ (OH) ₄		2θ	Int	h	k	l	2θ	Int	h	k	l
Aluminum Silicate Hydroxide		12.344	100	0	0	1	43.508*	5	0	2	3
		19.826*	35	0	2	0	43.863*	20	2	2	2
		20.340*	60	1	1	0	45.415*	35	2	0	3
Kaolinite-1A		21.225*	45	1	1	1	45.657*	35	1	3	2
Rad.: CuKα λ: 1.5418 Filter: Mono d-sp: 22.9		21.468*	35	1	1	1	45.975*	20	2	2	1
Cut off: Int.: Estimation L/Corr.:		23.120*	40	0	2	1	46.523*	20	2	2	1
Ref: Goodyear, Duffin, Mineral. Mag., 32, 902 (1961)		23.758*	25	0	2	1	46.853*	35	1	3	2
		24.877*	80	0	0	2	47.319*	20	0	4	2
		26.054*	5	1	1	1	47.715*	5	1	1	3
		26.400*	35	1	1	1	47.955*	25	1	3	3
Sys.: Triclinic S.G.: C1		28.286*	20	1	1	2	48.692*	20	0	4	2
a: 5.155 b: 8.959 c: 7.407 A: 0.5754 C: 0.8268		28.732*	20	1	1	2	49.396*	25	0	3	3
α: 91.68 β: 104.9 γ: 89.94 Z: 2 mp:		32.511*	20	0	2	2	49.596*	35	2	2	3
Ref: Ibid.		34.967*	35	2	0	1	50.417*	20	2	2	3
		35.151*	25	1	3	0	51.051*	25	0	0	4
		35.408*	35	1	3	1	53.592*	25	2	2	2
Dx: 2.595 Dm: 2.645 SS/FOM ₃ = 35(.022, 38)		35.641*	10	1	1	2	54.313*	25	2	1	5
		35.995*	45	2	0	0	54.593*	25	1	5	1
		37.717*	25	0	0	3	55.019*	40	2	2	4
		38.351*	40	2	0	2	55.343*	40	2	4	0
		38.504*	40	1	3	1	55.488*	10	2	4	3
sa: 1.559(6) naβ: 1.564(5) cr: 1.565(5) Sign: 2V:24-50		39.078*	5	1	1	3	55.744*	40	3	1	2
Ref: Deer, W., Howie, R., Zussman, J., Rock Forming Minerals, 3, 194		39.291*	35	1	3	1	56.338*	30	3	1	0
		40.018*	20	1	3	2	56.831*	70	1	3	3
		40.316*	5	0	4	0	57.333*	30	0	4	3
Color: White		40.677*	10	1	2	1	57.845*	10	1	3	5
Specimen from Scalby, Yorkshire, England, UK. Validated		41.083*	20	1	3	2	58.165*	60	1	3	4
by calculated pattern Borg and Smith, GSA Memoir, 122.		41.299*	20	2	0	1	58.733*	10	2	4	1
Kaolinite-serpentine group, dioctahedral subgroup.mC.D.		41.558*	5	2	2	0	59.523*	30	2	2	4
Cell: a=5.166, b=7.407, c=5.155, α=104.90, β=119.87.		42.003*	10	0	4	1	59.863*	40	1	1	4
γ=84.10, a/b=0.6974, c/b=0.6960, S.G.=P1(1), PSC: aP17.		42.374*	20	0	2	3	60.207*	40	2	0	3
To replace 5-143 and 12-447. Mwt: 258.16. Volume[CD]:		42.731*	10	0	4	1	61.219*	5	3	1	1
165.22.		43.224*	10	2	2	2	61.624*	5	2	4	3

2θ	Int	h	k	l
62.360*	90	3	3	1

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Quartz 05-0490

05-0490		Wavelength= 1.54056											
α-SiO ₂		2θ	Int	h	k	l	2θ	Int	h	k	l		
Silicon Oxide		20.835 [*]	35	1	0	0	81.186 [*]	4	1	1	4		
		26.643	100	1	0	1	81.486 [*]	4	3	1	0		
		36.526 [*]	12	1	1	0	83.836 [*]	2	3	1	1		
Quartz, low		39.455 [*]	12	1	0	2	84.941 [*]	<1	2	0	4		
Rad.: Cu λ: 1.5405 Filter: d-sp:		40.283 [*]	6	1	1	1	87.451 [*]	<1	3	0	3		
Cut off: Int.: I/cor.: 3.60		42.443 [*]	9	2	0	0	90.823 [*]	4	3	1	2		
Ref:		45.788 [*]	6	2	0	1	92.808 [*]	1	4	0	0		
		50.166 [*]	17	1	1	2	94.650 [*]	2	1	0	5		
		50.643 [*]	<1	0	0	3	95.127 [*]	2	4	0	1		
		54.864 [*]	7	2	0	2	96.236 [*]	2	2	1	4		
		55.330 [*]	3	1	0	3	98.747 [*]	2	2	2	3		
Sys.: Hexagonal S.G.: P3 ₁ 21 (152)		57.244 [*]	<1	2	1	0	102.224	2	1	1	5		
a: 4.913 b: a: 5.405 A: C: 1.1001		59.981 [*]	15	2	1	1	102.570	2	3	1	3		
α: β: γ: Z: 3 mp:		64.029 [*]	3	1	1	3	103.910	<1	3	0	4		
Ref: Ibid.		65.806 [*]	<1	3	0	0	104.195	1	3	2	0		
		67.748 [*]	7	2	1	2	106.603	2	3	2	1		
		68.140 [*]	11	2	0	3	112.206	<1	4	1	0		
		68.309 [*]	9	3	0	1							
Dx: 2.649 Dm: SS/FOM ₃ = 78(.0125, 31)		73.460 [*]	3	1	0	4							
		75.654 [*]	4	3	0	2							
		77.697 [*]	2	2	2	0							
PSC: hP9. To replace 1-649. Deleted by 33-1161. Mwt: 60.08. Volume[CD]: 112.98.		79.891 [*]	5	2	1	3							
		80.084 [*]	2	2	2	1							

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Quartz 46-1045

46-1045		Wavelength= 1.5405981											
SiO ₂		2θ	Int	h	k	l	2θ	Int	h	k	l		
Silicon Oxide		20.860 [*]	16	1	0	0	95.119 [*]	<1	4	0	1		
		26.640	100	1	0	1	96.238 [*]	1	2	1	4		
		36.544 [*]	9	1	1	0	98.751 [*]	1	2	2	3		
Quartz, syn		39.465 [*]	8	1	0	2	102.231	<1	1	1	5		
Rad.: CuKα1: 1.5405 Filter: Ge Mono d-sp: Diffractometer		40.300 [*]	4	1	1	1	102.567	<1	3	1	3		
Cut off: Int.: Diffract. I/cor.: 3.41		42.450 [*]	6	2	0	0	103.877	<1	3	0	4		
Ref: Kern, A., Eysel, W., Mineralogisch-Petrograph. Inst., Univ. Heidelberg, Germany, ICDD Grant-in-Aid, (1993)		45.793 [*]	4	2	0	1	104.203	<1	3	2	0		
		50.139 [*]	13	1	1	2	106.593	<1	3	2	1		
		50.622 [*]	<1	0	0	3	112.114	<1	4	1	0		
		54.875 [*]	4	2	0	2	114.061	<1	3	2	2		
		55.325 [*]	2	1	0	3	114.467	2	4	0	3		
Sys.: Hexagonal S.G.: P3 ₂ 21 (154)		57.235 [*]	<1	2	1	0	114.639	2	4	1	1		
a: 4.91344(4) b: c: 5.40524(8) A: C: 1.1001		59.960 [*]	9	2	1	1	115.885	<1	2	2	4		
α: β: γ: Z: 3 mp:		64.036 [*]	2	1	1	3	117.537	<1	0	0	6		
Ref: Ibid.		65.786 [*]	<1	3	0	0	118.313	<1	2	1	5		
		67.744 [*]	6	2	1	2	120.124	1	3	1	4		
		68.144 [*]	7	2	0	3	121.853	<1	1	0	6		
		68.318 [*]	5	3	0	1	122.605	<1	4	1	2		
Dx: 2.649 Dm: 2.660 SS/FOM ₃ = 539(.0018, 31)		73.468 [*]	2	1	0	4	127.251	<1	3	0	5		
		75.660 [*]	3	3	0	2	131.203	<1	1	1	6		
		77.675 [*]	1	2	2	0	132.756	<1	5	0	1		
ea: naδ: 1.544 σ: 1.553 Sign: ZV:		79.884 [*]	2	2	1	3	134.293	<1	4	0	4		
Ref: Swanson, Fuyat, Natl. Bur. Stand. (U.S.), Circ. 539, 3, 24 (1954)		80.047 [*]	<1	2	2	1	136.424	1	2	0	6		
		81.173 [*]	2	1	1	4	137.895	2	4	1	3		
		81.491 [*]	2	3	1	0	140.318	<1	3	3	0		
		83.840 [*]	1	3	1	1	143.251	3	5	0	2		
Color: White		84.957 [*]	<1	2	0	4	144.119	<1	3	3	1		
Integrated intensities. Pattern taken at 23(1) C. Low temperature quartz. 2θ determination based on profile fit method. O2 Si type. Quartz group. Silicon used as an internal stand. PSC: hP9. To replace 33-1161. Mwt: 60.08. Volume[CD]: 113.01.		87.439 [*]	<1	3	0	3							
		90.831 [*]	2	3	1	2							
		92.788 [*]	<1	4	0	0							
		94.651 [*]	1	1	0	5							

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Muscovite 07-0032

07-0032		Wavelength= 1.54056									
KAl ₂ Si ₃ AlO ₁₀ (OH) ₂		2θ	Int	h	k	l	2θ	Int	h	k	l
Potassium Aluminum Silicate Hydroxide		8.836<100	0	0	2		40.301*	6	0	4	1
		17.653* 35	0	0	4		40.971*	6	2	2	1
		19.801* 35	1	1	0		41.304*	8	0	2	8
Muscovite 2M1, syn		19.891* 65	1	1	1		42.008*	10	2	2	2
Rad.: Cu	λ: 1.5405	20.211* 14	0	2	1		42.359*	25	1	3	5
Filter:	d-sp:	20.639* 20	1	1	1		44.118*	6	0	4	4
Cut off:	Int.:	21.604* 14	0	2	2		45.067*	75	2	0	7
	I/cor.:	22.358* 12	1	1	2		45.911*	14	1	3	7
Ref:		22.848* 35	1	1	3		52.682*	6	2	2	8
		23.803* 30	0	2	3		53.921*	6	1	5	0
		25.427* 45	1	1	4		54.935*	12	0	0	12
Sys.: Monoclinic	S.G.: C2/m (12)	26.371<100	0	2	4		55.549*	18	1	3	9
a: 5.189	b: 8.995	27.786* 45	1	1	4		57.478*	8	1	1	12
c: 20.097	A: 0.5769	29.766* 45	0	2	5		61.843*	40	0	6	0
β: 95.18	γ:	31.126* 35	1	1	5						
Z: 4	mp:	31.901* 20	1	1	6						
Ref: Ibid.		34.617* 50	1	3	1						
		34.742* 45	1	1	6						
		34.994* 90	2	0	2						
Dx: 2.832	Dm:	35.684* 20	0	0	8						
	SS/FOM ₃ = 7(.063, 66)	36.526* 20	1	3	3						
		36.711* 12	2	0	2						
CAS #: 1318-94-1. C.D. Cell: a=20.097, b=8.995, c=5.189,		37.506* 10	2	0	4						
β=95.18, α/b=2.2342, c/b=0.5769, S.G.=A2/m(12). PSC:		37.767* 25	1	3	3						
mC84. Deleted by minerals in 1980. Mwt: 398.31.		40.096* 12	2	2	1						
Volume[CD]: 934.20.											

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Muscovite 07-0025

07-0025		Wavelength= 1.5418									
KAl ₂ Si ₃ AlO ₁₀ (OH) ₂		2θ	Int	h	k	l	2θ	Int	h	k	l
Potassium Aluminum Silicate Hydroxide		8.755* 100	0	0	1		17.597* 35	0	0	2	
		17.597* 35	0	0	2		19.773* 90	0	2	0	
Muscovite-1M, syn		20.416* 25	1	1	1		20.416* 25	1	1	1	
Rad.: CuKα	λ: 1.5418	21.622* 16	0	2	1		21.622* 16	0	2	1	
Filter: Ni Beta	M d-sp:	24.318* 60	1	1	2		24.318* 60	1	1	2	
Cut off:	Int.: Diffract.	26.52<100	0	0	3		26.52<100	0	0	3	
	I/cor.:	29.086* 30	1	1	2		29.086* 30	1	1	2	
Ref: Yoder, H., Eugster, H., Geochim. Cosmochim. Acta, 8, 225 (1955)		30.520* 6	1	1	3		30.520* 6	1	1	3	
		33.319* 16	0	2	3		33.319* 16	0	2	3	
Sys.: Monoclinic	S.G.: C2/m (12)	34.743* 50	1	3	0		34.743* 50	1	3	0	
a: 5.208	b: 8.995	34.981* 90	1	3	1		34.981* 90	1	3	1	
c: 10.275	A: 0.5790	35.193* 20	2	0	0		35.193* 20	2	0	0	
C: 1.1423		36.680* 12	1	3	1		36.680* 12	1	3	1	
α:	β: 101.6	37.391* 4	1	3	2		37.391* 4	1	3	2	
γ:	Z: 2	37.799* 12	1	1	4		37.799* 12	1	1	4	
mp:		40.148* 8	0	4	0		40.148* 8	0	4	0	
Ref: Ibid.		40.658* 8	2	2	0		40.658* 8	2	2	0	
		41.201* 4	0	4	1		41.201* 4	0	4	1	
Dx: 2.805	Dm: 2.825	41.901* 20	1	3	3		41.901* 20	1	3	3	
	SS/FOM ₂₅ = 9(.037, 84)	42.880* 6	2	0	2		42.880* 6	2	0	2	
α: 1.563(11)αβ: 1.596(14)γ: 1.602(15)δ: 2V:30-47		45.034* 30	0	0	5		45.034* 30	0	0	5	
Ref: Ibid.		46.397* 8	1	3	3		46.397* 8	1	3	3	
		47.875* 4	1	3	4		47.875* 4	1	3	4	
		55.054* 18	2	4	2		55.054* 18	2	4	2	
Color: Colorless, light green, pink, light brown		55.597* 12	1	5	1		55.597* 12	1	5	1	
CAS #: 1318-94-1. Synthesized from K Al Si O ₄ + kaolinite + water at 200 C and 15,000 p.s.i. for 4120 hours.		56.263* 12	2	0	4		56.263* 12	2	0	4	
Mica group, dioctahedral subgroup. C.D. Cell: a=10.275, b=8.995, c=5.208, β=101.60, α/b=1.1423, c/b=0.5790.		61.219* 4	1	3	5		61.219* 4	1	3	5	
S.G.=A2/m(12). PSC: mC42. To replace 2-56. Mwt: 398.31.		61.898* 35	0	6	0		61.898* 35	0	6	0	
Volume[CD]: 471.51.											

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Gibbsite 33-0018

33-0018		Wavelength= 1.54060									
Al(OH) ₃		2 θ	Int	h	k	l	2 θ	Int	h	k	l
Aluminum Hydroxide		18.283	100	0	0	2	59.494*	3	2	0	NO
		20.300*	70	1	1	0	62.512*	4	2	1	NO
		20.349*	50	2	0	0	62.512*	4	2	3	2
Gibbsite, syn		26.515*	17	2	0	3	63.800*	30	3	2	4
Rad.: CuK α 1: 1.5406 Filter: d-sp: Diffractometer		26.896*	30	1	1	3	63.800*	30	3	3	4
Cut off: Int.: Diffract. I/cor.: 1.0		28.011*	25	1	1	2	64.653*	18	4	1	NO
Ref: Cisar, A., Poulsen, K., Dow Chemical Company, Freeport, TX, USA, ICDD Grant-in-Aid, (1979)		28.725*	13	1	0	3	66.149*	19	3	3	NO
		36.407*	25	3	1	1	66.650*	13	3	1	NO
		36.616*	40	0	2	1	67.817*	6	3	3	NO
		37.083*	15	0	0	4	68.883*	10	0	2	NO
		37.664*	55	3	1	1	70.718*	7	1	2	NO
Sys.: Monoclinic S.G.: P2 ₁ /n (14)		38.318*	4	1	2	1	71.111*	4	2	2	NO
a: 8.6552 b: 5.0722 c: 9.7161 A: 1.7064 C: 1.9156		39.314*	15	3	1	2	72.681*	3	4	1	NO
α : β : 94.607 γ : Z: 8 mp:		40.108*	20	0	2	2	77.430*	3	6	2	NO
Ref: Ibid.		41.140*	2	1	2	3	77.797*	2	3	3	4
		41.691*	27	3	1	2	78.613*	6	4	2	5
		43.374*	4	1	1	4	78.613*	6	2	4	0
		44.167*	40	3	1	3	78.954*	7	1	4	NO
Dx: 2.437 Dm: 2.400 SS/FOM ₃ Σ =10(.025,113)		44.754*	3	2	2	2	81.581*	4	1	1	NO
		45.440*	28	0	2	3	84.541*	4	6	2	NO
cc: 1.577 no β : 1.577 sp: 1.595 Sign: 2V:0		46.192*	6	1	2	3	89.589*	3	4	4	NO
Ref: Dana's System of Mineralogy, 7th Ed., I, 663 (1944)		46.192*	6	2	2	2	90.719*	4	6	3	NO
		47.306*	15	1	2	3					
		50.548*	30	3	2	1					
		52.175*	30	0	2	4					
Color: White		52.667*	4	1	2	4					
Sample of reagent grade chemical from Matheson, Coleman, and Bell. Optical data on artificial material; measured density on crystals. C.D. Cell: a=9.716, b=5.072, c=8.655, β =94.61, a/b=1.9156, c/b=1.7064, S.G.=P2 ₁ /n(14). Silicon used as an internal stand. PSC: mP36. To replace 1-263, 1-264, 1-265, 1-266, 7-324 and 12-460 and validated by calculated pattern 29-41. Mwt: 78.00. Volume[CD]: 425.17.		53.977*	4	1	2	4					
		54.424*	30	3	1	4					
		55.383*	9	1	3	0					
		55.383*	9	2	2	4					
		57.851*	7	4	1	4					
		58.095*	7	3	1	0					
		58.605*	8	5	0	5					
		58.605*	8	2	3	0					

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Gibbsite 07-0324

07-0324		Wavelength= 1.5418									
Al(OH) ₃		d Å	Int	h	k	l	d Å	Int	h	k	l
Aluminum Hydroxide		4.8500	100	0	0	2	1.39800	2	3	1	6
		4.37000	16	1	1	0	1.38000	1	1	0	7
		4.32000	8	2	0	0	1.36100	1	0	2	6
Gibbsite		3.30600	5	1	1	2	1.34000	1	4	0	6
Rad.: CuK α λ : 1.5418 Filter: Ni Beta M d-sp:		3.18700	4	1	1	2	1.33000	1	4	3	0
Cut off: Int.: Diffract. I/cor.:		3.11200	3	2	0	2	1.32000	1	3	1	6
Ref: Gillery, Min. Ind., Penn State University, University Park, Pennsylvania, USA, Private Communication		2.45400	8	0	2	1	1.24600	1	1	4	1
		2.42000	6	0	0	4	1.21400	1	0	2	7
		2.38800	8	3	1	1					
		2.28500	2	3	1	2					
Sys.: Monoclinic S.G.: P2 ₁ /n (14)		2.24400	3	2	1	3					
a: 8.659 b: 5.077 c: 9.703 A: 1.7055 C: 1.9112		2.16800	3	3	1	2					
α : β : 94.2 γ : Z: 8 mp:		2.08500	1	1	1	4					
Ref: Ibid.		2.04300	6	3	1	3					
		1.99300	4	0	2	3					
		1.96000	1	1	2	3					
		1.92100	4	4	1	1					
		1.79900	4	3	1	4					
Dx: 2.436 Dm: 2.400 SS/FOM ₃ Σ =7(.031,146)		1.75000	5	0	2	4					
		1.68900	4	3	1	4					
cc: 1.568 no β : 1.568 sp: 1.587 Sign: 2V:0		1.65400	1	2	2	4					
Ref: Dana's System of Mineralogy, 7th Ed.		1.63800	1	4	2	1					
		1.59300	1	2	2	4					
		1.58400	1	4	2	2					
		1.57300	1	3	0	3					
Color: Colorless		1.55500	1	4	0	4					
Specimen from Cheener mine, Richmond, Massachusetts, USA. C.D. Cell: a=9.703, b=5.077, c=8.659, β =94.20, a/b=1.9112, c/b=1.7055, S.G.=P2 ₁ /n(14). PSC: mP36. To replace 1-263, 1-264, 1-265 and 1-266. Deleted by 33-18, lower FN, MTG 5/92. Mwt: 78.00. Volume[CD]: 425.42.		1.55100	1	2	3	1					
		1.48600	1	3	1	5					
		1.47700	1	3	2	5					
		1.45700	3	3	3	0					
		1.44100	2	6	0	0					
		1.40900	2	6	0	2					

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Montmorillonite 13-0135

13-0135		Wavelength= 1.5418				
<chem>Ca0.2(Al,Mg)2Si4O10(OH)2.4H2O</chem>		2 θ	Int	h	k	l
Calcium Magnesium Aluminum Silicate Hydroxide Hydrate		5.892°	100	0	0	1
		17.703°	60	0	0	3
		19.728°	80	1	0	0
Montmorillonite-15A		23.598°	20	0	0	4
Rad.:	λ : Filter: d-sp: Diffractometer	25.448°	10			
Cut off:	Int.: Diffraction I/cor.:	27.019°	10	1	0	3
Ref: Rosenquist, Nor. Geol. Tidsskr., 39, 350 (1959)		29.579°	60	0	0	5
		34.771°	40	1	1	0
		35.921°	40	0	0	6
		39.889°	10	2	0	0
		42.023°	10	0	0	7
Sys.: Hexagonal	S.G.: P	48.416°	10	0	0	8
a: 5.169(7)	b: c: 15.02(3) A: C: 2.9058	53.933°	30	2	1	0
α :	β : γ : Z: 1 mp:	61.852°	50	0	0	10
Ref: Bayliss, P., Powder Diffraction, 4, 19 (1989)		62.175°	50	3	0	0
		73.729°	20	2	2	1
		76.660°	20	3	1	0
Dx:	Dm: 2.300 SS/FOM ₁ ² =2(.127,66)					
ω :	ω : 1.545(6) ω : 1.57(7) ω : 1.57(7) Sign: 2V:0(15)					
Ref: Ibid.						
Color: White, yellow, green						
Specimen from Skyrvedalen, Hemsedal, Norway. CAS #: 1318-93-0. Analysis (wt.%): Si O ₂ 59.58, Al ₂ O ₃ 22.96, Fe ₂ O ₃ 0.47, MgO 3.67, CaO 3.38, Na ₂ O 0.06, loss 1* 9.61. Smectite group, dioctahedral subgroup.						
* 15.0 expands to 18.0 with glycerol treatment.						
PSC: hP32.20. Volume[CD]: 347.55.						
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Halloysite 09-0451

09-0451		Wavelength= 1.78897				
<chem>Al2Si2O5(OH)4.2H2O</chem>		2 θ	Int	h	k	l
Aluminum Silicate Hydroxide Hydrate		10.162°	90	0	0	1
		23.351°	100	1	0	0
		31.068°	90	0	0	3
		40.902°	80	1	1	0
Rad.:	λ : Filter: d-sp:	44.546°	60			
Cut off:	Int.: Estimation I/cor.:	47.296°	20	2	0	0
Ref: MacEwan, Amoros, Anales Edafol. Fisol. Vegetal (Madrid), 9, 363 (1950)		51.471°	10			
		64.426°	80	2	1	0
		74.310°	90	3	0	0
		88.403°	70	2	2	0
Sys.: Hexagonal	S.G.: P	93.111°	70	3	1	0
a: 5.122(4)	b: c: 10.03(11) A: C: 1.9582	108.092°	40	3	1	4
α :	β : γ : Z: 1 mp:	122.348°	20	1	1	9
Ref: Bayliss, P., Powder Diffraction, 4, 19 (1989)		136.513°	20	4	1	1
		"	40	3	3	0
		"	20	4	2	0
Dx:	Dm: 2.144 SS/FOM ₁ ² =1(.112,116)					
ω :	ω : 1.490 ω : Sign: 2V:					
Ref: Alexander, L et al., Am. Mineral., 28, 1 (1943)						
Color: Colorless						
Kaolinite-serpentine group, dioctahedral subgroup. Also called: endellite. PSC: hP23. Mwt: 294.19. Volume[CD]: 227.88.						
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Mullite 15-0776

15-0776		Wavelength= 1.54056									
Al ₆ Si ₂ O ₁₃		2 θ	Int	h	k	l	2 θ	Int	h	k	l
Aluminum Silicate		16.432°	50	1	1	0	66.514°	8	5	2	0
		23.354°	8	2	0	0	67.130°	<2	1	1	2
		25.971°	95	1	2	0	69.616°	6	2	0	2
Mullite, syn		26.267°	100	2	1	0	69.806°	6	4	4	0
Rad.: CuK α 1: 1.5405 Filter: Ni Beta M d-sp:		30.960°	20	0	0	1	70.442°	12	1	5	1
Cut off: Int.: Diffract. I/Cor.:		33.228°	40	2	2	0	70.844°	5	1	2	2
Ref: Natl. Bur. Stand. (U.S.) Monogr. 25, 3, 3 (1964)		35.278°	50	1	1	1	70.991°	5	2	1	2
		36.993°	14	1	3	0	71.576°	4	5	1	1
		37.354°	<2	3	1	0	71.904°	3	3	5	0
		38.992°	4	0	2	1	72.647°	4	5	3	0
		39.276°	20	2	0	1	73.901°	7	0	6	0
Sys.: Orthorhombic S.G.: Pbam (55)		40.874°	60	1	2	1	74.191°	13	2	5	1
a: 7.5456 b: 7.6898 c: 2.8842 A: 0.9812 C: 0.3751		42.590°	25	2	3	0	74.580°	6	2	2	2
α : β : γ : Z: .75 mp:		42.908°	8	3	2	0	75.162°	12	5	2	1
Ref: Ibid.		46.059°	2	2	2	1	75.555°	<2	6	0	0
		47.227°	2	0	4	0	76.836°	6	1	3	2
		48.184°	8	4	0	0	77.182°	2	3	1	2
		48.845°	<2	1	4	0	78.311°	2	4	4	1
Dx: 3.171 Dm: 3.000 SS:FOM ₃ =60(.0135, 37)		49.468°	10	3	1	1	78.835°	<2	2	6	0
		50.812°	<2	3	3	0	80.480°	4	2	3	2
ca: 1.637 η op: 1.641 η r: 1.652 Sign+ 2V:45-50		53.462°	6	2	4	0	81.046°	3	5	3	1
Ref: Winchell, Elements of Optical Mineralogy, 2, 401		53.883°	14	3	2	1	84.493°	<2	4	0	2
		54.093°	10	4	2	0	87.002°	1	2	6	1
		57.561°	20	0	4	1	88.569°	4	2	4	2
		58.412°	12	4	0	1	89.090°	5	4	2	2
Color: Colorless		58.994°	2	1	4	1	93.817°	<2	2	7	0
Pattern taken at 25 C. Sample was prepared from stoichiometric mixture of Al ₂ O ₃ and SiO ₂ 1x H ₂ O.		59.763°	2	4	1	1	98.446°	4	1	7	1
Sample was repeatedly ground and heated up to temperature of 1725 C. Spectrographic analysis: 0.01 to 0.1% Fe, and 0.001 to 0.01% each of Ca, Cr, Mg, Mn, Ni, Tl and Zr. Chemical analysis showed Al ₂ O ₃ 61.6, SiO ₂ 38 (mole%). Tungsten used as an internal stand. PSC: 0P15.75. Mwt: 426.05. Volume[CD]: 167.35.		60.711°	35	3	3	1	98.958°	4	2	5	2
		61.492°	<2	1	5	0	99.868°	8	3	7	0
		62.674°	<2	5	1	0					
		63.054°	<2	2	4	1					
		63.661°	8	4	2	1					
		64.571°	18	0	0	2					
		65.494°	4	2	5	0					

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Sillimanite 38-0471

38-0471		Wavelength= 1.54178									
Al₂SiO₅		2 θ	Int	h	k	l	2 θ	Int	h	k	l
Aluminum Silicate		16.520*	12	1	1	0	49.060*	1	1	3	2
		19.401*	2	1	0	1	49.740*	2	3	1	2
		23.168*	14	0	2	0	50.135*	1	0	4	1
Sillimanite		23.771*	3	2	0	0	50.135*	1	4	1	0
Rad.: CuK α λ : 1.5417 Filter: Graph Mono d-sp: Diffractometer		26.092	100	1	2	0	51.133*	2	3	3	0
Cut off: 17.7 Int.: Diffract. I/Cor.:		26.479*	35	2	1	0	51.660*	1	1	4	1
Ref: Keller, L., Rask, J., Buseck, P., Arizona State Univ., Tempe, AZ, USA, ICDD Grant-in-Aid, (1987)		27.915*	1	0	2	1	53.693*	7	2	4	0
		30.413*	1	1	2	1	53.693*	7	3	3	1
		30.756*	1	2	1	1	54.146*	2	3	2	2
		30.985*	7	0	0	2	54.554*	3	4	2	0
Sys.: Orthorhombic S.G.: Pbnm (62)		33.434*	16	2	2	0	54.751*	1	1	2	3
a: 7.486(1) b: 7.675(1) c: 5.7729(6) A: 0.9754 C: 0.7522		35.307*	20	1	1	2	54.968*	1	2	1	3
α :		36.992*	3	2	2	1	57.678*	12	0	4	2
Ref: Ibid.		37.135*	20	1	3	0	58.802*	1	4	0	2
		37.914*	1	3	1	0	59.108*	3	2	2	3
		39.042*	2	0	2	2	59.108*	3	1	4	2
		39.362*	3	3	0	1	60.782*	1	3	0	3
		39.362*	3	2	0	2	60.950*	14	3	4	0
Dx: 3.245 Dm: 3.250 SS/FOM $\bar{3}$ =50(0.159, 38)		40.391*	1	1	3	1	60.950*	14	3	3	2
		40.946*	30	1	2	2	61.375*	1	4	3	0
		41.161*	2	3	1	1	61.683*	1	1	5	0
		41.161*	2	2	1	2	62.095*	1	3	1	3
Ref: Deer, W., Howie, R., Zussmann, J., Rock Forming Minerals, 1, 121 (1962)		42.816*	12	2	3	0	63.277*	1	2	4	2
		43.246*	1	3	2	0	63.277*	1	5	1	0
		45.739*	1	2	3	1	63.678*	1	4	3	1
Color: Colorless		46.189*	1	3	2	1	64.021*	1	1	5	1
Specimen from Norwich, CT, USA. Chemical analysis, average of seven (wt.%): Si O ₂ 36.15, Al ₂ O ₃ 62.17, Fe ₂ O ₃ 0.90. Silicon used as an internal stand. PSC: oP32. To replace 10-369 and 22-18. Mwt: 162.05. Volume[CD]: 331.68.		46.189*	1	2	2	2	64.021*	1	4	2	2
		48.650*	2	4	0	0	64.269*	1	5	0	1
		48.867*	1	1	0	3	64.563*	5	0	0	4
		49.060*	1	1	4	0	65.588*	1	5	1	1

2 θ	Int	h	k	l	2 θ	Int	h	k	l
65.588*	1	2	3	3	83.882*	1	0	4	4
65.744*	4	2	5	0	84.612*	1	6	0	2
65.954*	1	3	2	3	84.840*	1	4	0	4
67.136*	2	5	2	0	84.840*	1	2	5	3
67.136*	2	1	1	4	85.107*	2	1	4	4
67.971*	1	2	5	1	87.322*	1	3	6	1
69.591*	1	0	2	4	87.322*	1	2	6	2
70.306*	2	4	4	0	88.759*	1	2	4	4
70.306*	2	4	3	2	89.035*	1	4	4	3
70.427*	2	1	4	3	89.230*	3	4	5	2
70.633*	8	1	5	2	89.230*	3	6	2	2
70.884*	5	1	2	4	89.468*	1	4	2	4
71.075*	3	2	1	4	89.780*	1	5	4	2
72.135*	1	5	1	2	89.780*	1	2	1	5
72.135*	1	3	3	3					
72.256*	1	3	5	0					
72.410*	1	4	4	1					
74.118*	30	0	6	0					
74.417*	8	3	5	1					
74.417*	8	2	5	2					
74.704*	2	2	2	4					
74.988*	1	4	2	3					
75.372*	2	5	3	1					
75.372*	2	1	6	0					
75.732*	2	5	2	2					
76.275*	1	0	6	1					
76.275*	1	6	0	0					
76.908*	1	1	3	4					
77.389*	1	3	1	4					
77.493*	1	1	6	1					
77.493*	1	6	1	0					
78.747*	1	4	4	2					
79.113*	1	2	6	0					
80.647*	1	2	3	4					
80.647*	1	3	5	2					
80.917*	1	4	3	3					
80.917*	1	3	2	4					
81.181*	1	1	5	3					
81.181*	1	2	6	1					
81.590*	1	5	4	0					
81.590*	1	5	3	2					
82.472*	1	0	6	2					

APPENDIX C

True density (specific gravity) and particle size distribution data

Table 1C True density of raw materials

True density*		RC6	RC9	RC10	RC11	RC12
(g/cm ³)						
BCW	2.5174	40%	40%	60%	60%	60%
B85	2.6109	-	60%	-	20%	40%
K325	2.6164	60%	-	40%	20%	-
Calculated true density (g/cm ³)		2.5735	2.5735	2.5570	2.5559	2.5548

* True density of Hypure Vector[®] = 2.5715 g/cm³

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Table 2C Particle size distribution of raw materials compared with Hypure Vector®. (refer to Fig. 5.6)

Equivalent spherical diameter (μm)	Weight percent finer			
	VECTOR	BCW	K325	B85
40.0	100.0	100.0	100.0	100.0
30.0	100.0	100.0	97.0	93.6
20.0	100.0	100.0	93.4	85.9
10.0	94.7	88.4	67.6	59.8
8.0	94.7	83.5	59.2	52.4
6.0	91.2	77.9	49.9	44.1
5.0	88.3	74.2	44.0	40.1
4.0	84.4	69.5	37.5	34.8
3.0	77.0	64.0	31.6	29.2
2.0	65.6	57.0	24.2	22.5
1.0	42.6	44.6	13.8	13.8
0.8	35.2	40.3	11.1	11.2
0.6	26.2	34.3	8.1	8.3
0.5	21.1	30.2		6.6
0.4	15.7	24.7		
0.3		17.3		

Table 3C Particle size distribution of selected RC formula (refer to Fig. 5.12)

Equivalent spherical diameter (μm)	Weight percent finer					
	VECTOR	RC6	RC9	RC10	RC11	RC12
40.0	100.0	100.0	100.0	100.0	100.0	100.0
30.0	100.0	85.3	100.0	100.0	83.5	100.0
20.0	100.0	73.0	92.0	86.4	81.3	92.5
10.0	94.7	58.9	71.3	73.4	64.8	74.8
8.0	94.7	52.9	64.2	69.1	56.6	68.0
6.0	91.2	46.3	56.7	62.2	50.0	60.5
5.0	88.3	43.1	53.0	58.1	45.9	56.1
4.0	84.4	37.5	45.6	51.8	43.1	49.8
3.0	77.0	31.4	40.1	45.1	37.1	42.6
2.0	65.6	25.4	33.5	38.0	31.2	35.6
1.0	42.6	16.4	23.5	26.7	21.9	25.7
0.8	35.2	14.0	20.4	23.2	19.3	22.5
0.6	26.2	10.9	16.3	18.8	15.9	18.5
0.5	21.1	9.2		16.0	13.8	15.8
0.4	15.7	7.0		12.4	11.1	12.3
0.3				8.2	8.0	8.1

Table 4C Particle size distribution of slip before spray drying
(refer to Fig. 5.26)

Equivalent spherical diameter (μm)	Weight percent finer					
	VECTOR	40RC11 +1B	50RC11 +1B	50RC9 +1B	60RC9	60RC9 +1B
40.0	100.0	100.0	100.0	100.0	100.0	100.0
30.0	100.0	97.7	88.8	100.0	100.0	100.0
20.0	100.0	89.5	73.6	79.3	83.1	92.9
10.0	94.7	66.6	56.4	57.2	64.9	70.2
8.0	94.7	62.3	51.2	51.2	58.8	64.3
6.0	91.2	56.6	45.0	43.8	53.7	55.1
5.0	88.3	52.6	41.0	39.7	49.8	49.5
4.0	84.4	47.9	37.0	37.3	44.7	43.2
3.0	77.0	43.2	32.8	32.8	39.8	39.2
2.0	65.6	37.6	27.3	28.2	36.3	33.8
1.0	42.6	28.5	20.2	21.9	28.9	25.9
0.8	35.2	26.0	18.0	20.3	26.6	23.9
0.6	26.2	23.2	15.0	18.3	23.9	21.5
0.5	21.1	21.7	13.1	17.3	22.1	20.1
0.4	15.7	20.2	10.7	16.1	20.2	18.8
0.3		18.8	7.8		18.1	

APPENDIX D

Deflocculant demand and shear response data

Table 1D Effect of deflocculant content on viscosity of slips at 50 wt% solid compared with Hypure Vector[®]. (refer to Fig. 5.11)

%Na ₂ SiO ₃ by dry wt.	Viscosity(cP) of slip at 50% solid after adding Na ₂ SiO ₃					
	50RC6	50RC9	50RC10	50RC11	50RC12	Vector
0.02						87.0
0.03		165.0				84.8
0.04		143.8				82.5
0.05	217.5	122.5				77.5
0.06	147.5	101.3				71.5
0.07	117.5	80.0			462.5	65.0
0.08	97.5	72.5	492.5	352.5	392.5	59.5
0.09	85.0	65.0	408.8	301.3	322.5	54.0
0.10	80.0	57.5	325.0	250.0	283.8	53.0
0.11	75.0	56.9	301.7	233.3	245.0	52.0
0.12	70.0	56.3	278.3	216.7	229.2	51.0
0.13	62.5	55.6	255.0	200.0	213.3	46.0
0.14	62.5	55.0	228.8	186.3	197.5	41.0
0.15	55.0	52.5	202.5	172.5	182.5	43.0
0.16	55.0	50.0	190.0	161.3	167.5	45.0
0.17		47.5	177.5	147.0	157.5	42.8
0.18		46.3	171.7	145.0	147.5	40.7
0.19		45.0	165.8	140.0	141.7	38.5
0.20		43.8	160.0	135.0	135.8	
0.21		42.5	151.3	130.0	130.0	
0.22		41.7	142.5	125.0	125.0	
0.23		40.8	136.3	120.0	120.0	
0.24		40.0	130.0	115.0	110.0	
0.25		39.4	123.8	110.8	110.0	
0.26		38.8	117.5	106.7	105.0	
0.27		38.1	114.2	102.5	100.0	
0.28		37.5	110.8	98.8	96.7	
0.29		36.7	107.5	95.0	93.3	
0.30		35.8	102.5		90.0	
0.31		35.0	97.5		87.5	
0.32		35.8	95.0		85.0	
0.33		36.7	92.5			
0.35		37.5				

Table 2D Effect of deflocculant content on viscosity of RC11 slip at 40 and 50 wt% solid with and without addition of binder (1 wt%).

(refer to Fig. 5.16)

%Na ₂ SiO ₃ by dry wt.	Viscosity (cP) of RC11 slips after adding Na ₂ SiO ₃			
	40RC11	40RC11+1B	50RC11	50RC11+1B
0.00	67.5			
0.02	52.5			
0.03	40.0			
0.04	36.3	262.5		
0.05	32.5	183.8	352.5	
0.06	35.0	105.0	301.3	
0.07	32.5	85.0	250.0	
0.08	30.0	65.0	200.0	
0.09	27.5	60.0	186.3	332.5
0.10	30.0	55.0	172.5	278.8
0.11	32.5	55.0	161.3	225.0
0.12		55.0	150.0	145.0
0.13			135.0	133.8
0.14			130.0	122.5
0.15			125.0	100.0
0.16			115.0	92.5
0.17			108.8	85.0
0.18			102.5	82.5
0.19			95.0	80.0
0.20				75.0
0.22				70.0
0.24				70.0

Table 3D Effect of deflocculant content on viscosity of RC9 slip at 50 and 60 wt% solid with and without addition of binder (1 wt%).

(refer to Fig. 5.17, Fig 5.18)

%Na ₂ SiO ₃ By dry wt.	Viscosity(cP) after adding Na ₂ SiO ₃		%Na ₂ SiO ₃ By dry wt.	Viscosity(cP) after adding Na ₂ SiO ₃	
	50RC9	50RC9+1B		60RC9	60RC9+1B
0.00			0.23	480.0	
0.03	165.0	450.0	0.25	377.5	
0.05	136.7	310.0	0.28	317.5	
0.06	108.3	170.0	0.30	280.0	490.0
0.07	80.0	135.0	0.33	250.0	443.8
0.08	72.5	100.0	0.35	225.0	397.5
0.09	65.0	85.0	0.40	197.5	340.0
0.10	57.5	70.0	0.45	172.5	305.0
0.11	56.7	66.7	0.50	157.5	280.0
0.12	55.8	63.3	0.55	142.5	265.0
0.13	55.0	60.0	0.60	140.0	247.5
0.15	51.3	52.5	0.65	132.5	235.0
0.17	47.5	47.5	0.70	127.5	222.5
0.19	45.0	45.0	0.75	125.0	217.5
0.20	42.5	43.8	0.78	125.0	212.5
0.22	41.3	42.5	0.80	125.0	208.8
0.23	40.0	42.5	0.85	122.5	205.0
0.24	39.2	42.5	0.90	120.0	200.0
0.26	38.3	40.0	0.93		195.0
0.27	37.5		1.00		190.0
0.30	35.0		1.08		185.0
0.33	37.5		1.15		180.0
			1.23		177.5

Table 4D Variation of shear stress with shear rate
(refer to Fig. 5.19, Fig 5.21)

rpm	Small sample DV II+ Spindle No.18							
	40% RC11 + 1 wt% Binder + 0.08 wt% Na ₂ SiO ₃							
	η -up (Pa.s)	$\dot{\gamma}$ (sec ⁻¹)	τ -up (Pa)	τ -down (Pa)	log ($\dot{\gamma}$)	log (τ) up	log (τ) down	log (η)
0.3	0.15	0.396	0.06	0.29	-0.40	-1.23	-0.54	-0.83
0.6	0.18	0.792	0.15	0.38	-0.10	-0.84	-0.42	-0.73
1.5	0.16	1.980	0.32	0.53	0.30	-0.50	-0.28	-0.80
3	0.13	3.960	0.52	0.68	0.60	-0.28	-0.17	-0.88
6	0.10	7.920	0.78	0.86	0.90	-0.11	-0.07	-1.00
12	0.07	15.800	1.13	1.13	1.20	0.05	0.05	-1.15
30	0.04	39.600	1.77	1.73	1.60	0.25	0.24	-1.35
60	0.03	79.200	2.09	2.09	1.90	0.32	0.32	-1.58
Stop 5 min								
0.3	3.89	0.396	1.54	1.54	-0.40	0.19	0.19	0.59
rpm	Small sample DV II+ Spindle No.31							
	50% RC11 + 1 wt% Binder + 0.14 wt% Na ₂ SiO ₃							
	η -up (Pa.s)	$\dot{\gamma}$ (sec ⁻¹)	τ -up (Pa)	τ -down (Pa)	log ($\dot{\gamma}$)	log (τ) up	log (τ) down	log (η)
0.3	0.40	0.102	0.04	3.40	-0.99	-1.39	0.53	-0.40
0.6	6.02	0.204	1.23	3.43	-0.69	0.09	0.54	0.78
1.5	5.29	0.510	2.70	3.77	-0.29	0.43	0.58	0.72
3.0	3.36	1.020	3.43	3.92	0.01	0.54	0.59	0.53
6.0	1.96	2.040	4.00	4.12	0.31	0.60	0.61	0.29
12.0	1.17	4.080	4.78	4.62	0.61	0.68	0.66	0.07
30.0	0.56	10.200	5.76	5.60	1.01	0.76	0.75	-0.25
60.0	0.33	20.400	6.76	6.76	1.31	0.83	0.83	-0.48
Stop 5 min								
0.3	15.10	0.102	1.54	1.54	-0.99	0.19	0.19	1.18
rpm	Small sample DV II+ Spindle No.31							
	50% RC9 + 0.10 wt% Na ₂ SiO ₃							
	η -up (Pa.s)	$\dot{\gamma}$ (sec ⁻¹)	τ -up (Pa)	τ -down (Pa)	log ($\dot{\gamma}$)	log (τ) up	log (τ) down	log (η)
0.3	0.20	0.102	0.02	0.06	-0.99	-1.70	-1.21	-0.71
0.6	0.15	0.204	0.03	0.07	-0.69	-1.51	-1.15	-0.82
1.5	0.16	0.510	0.08	0.13	-0.29	-1.09	-0.88	-0.79
3	0.14	1.020	0.14	0.20	0.01	-0.84	-0.69	-0.85
6	0.12	2.040	0.25	0.29	0.31	-0.61	-0.54	-0.92
12	0.10	4.080	0.40	0.43	0.61	-0.40	-0.37	-1.01
30	0.07	10.200	0.73	0.76	1.01	-0.13	-0.12	-1.14
60	0.06	20.400	1.15	1.15	1.31	0.06	0.06	-1.25
Stop 5 min								
0.3	15.10	0.102	1.54	1.54	-0.99	0.19	0.19	1.18

Table 5D Variation of shear stress with shear rate
(refer to Fig. 5.22, Fig 5.24)

rpm	Small sample DV II+ Spindle No.31							
	50% RC9 + 1 wt% Binder + 0.06 wt% Na ₂ SiO ₃							
	η -up (Pa.s)	$\dot{\gamma}$ (sec ⁻¹)	τ -up (Pa)	τ -down (Pa)	log ($\dot{\gamma}$)	log (τ) up	log (τ) down	log (η)
0.3	3.20	0.102	0.33	2.14	-0.99	-0.49	0.33	0.50
0.6	3.60	0.204	0.73	2.14	-0.69	-0.13	0.33	0.56
1.5	3.14	0.510	1.60	2.21	-0.29	0.20	0.34	0.50
3	2.01	1.020	2.05	2.26	0.01	0.31	0.35	0.30
6	1.22	2.040	2.48	2.50	0.31	0.39	0.40	0.08
12	0.73	4.080	2.97	2.87	0.61	0.47	0.46	-0.14
30	0.36	10.200	3.68	3.50	1.01	0.57	0.54	-0.44
60	0.22	20.400	4.41	4.41	1.31	0.64	0.64	-0.67
Stop 5 min								
0.3	15.10	0.102	1.54	1.54	-0.99	0.19	0.19	1.18
rpm	Small sample DV II+ Spindle No.31							
	60% RC9 + 0.55 wt% Na ₂ SiO ₃							
	η -up (Pa.s)	$\dot{\gamma}$ (sec ⁻¹)	τ -up (Pa)	τ -down (Pa)	log ($\dot{\gamma}$)	log (τ) up	log (τ) down	log (η)
0.3	1.10	0.102	0.11	0.16	-0.99	-0.95	-0.79	0.04
0.6	0.80	0.204	0.16	0.20	-0.69	-0.79	-0.69	-0.10
1.5	0.56	0.510	0.29	0.31	-0.29	-0.54	-0.51	-0.25
3.0	0.44	1.020	0.45	0.47	0.01	-0.35	-0.33	-0.36
6.0	0.33	2.040	0.67	0.68	0.31	-0.17	-0.17	-0.48
12.0	0.27	4.080	1.09	1.08	0.61	0.04	0.03	-0.57
30.0	0.20	10.200	2.08	2.06	1.01	0.32	0.31	-0.69
60.0	0.17	20.400	3.42	3.42	1.31	0.53	0.53	-0.78
Stop 5 min								
0.3	15.10	0.102	1.54	1.54	-0.99	0.19	0.19	1.18
rpm	Small sample DV II+ Spindle No.31							
	60% RC9 + 1 wt% Binder + 0.80 wt% Na ₂ SiO ₃							
	η -up (Pa.s)	$\dot{\gamma}$ (sec ⁻¹)	τ -up (Pa)	τ -down (Pa)	log ($\dot{\gamma}$)	log (τ) up	log (τ) down	log (η)
0.3	1.70	0.102	0.17	0.21	-0.99	-0.76	-0.67	0.23
0.6	1.35	0.204	0.28	0.29	-0.69	-0.56	-0.54	0.13
1.5	0.84	0.510	0.43	0.51	-0.29	-0.37	-0.29	-0.08
3	0.64	1.020	0.65	0.76	0.01	-0.19	-0.12	-0.19
6	0.51	2.040	1.04	1.14	0.31	0.02	0.06	-0.29
12	0.42	4.080	1.72	1.81	0.61	0.24	0.26	-0.38
30	0.33	10.200	3.36	3.38	1.01	0.53	0.53	-0.48
60	0.27	20.400	5.45	5.45	1.31	0.74	0.74	-0.57
Stop 5 min								
0.3	15.10	0.102	1.54	1.54	-0.99	0.19	0.19	1.18

Table 6D Variation of shear stress with shear rate of 40%RC11 and 50%RC11 with 1 wt% binder (refer to Fig. 1D)

rpm	Small sample DV II+ Spindle No.18					
	40% RC11 + 1 wt% Binder + 0.08 wt% Na ₂ SiO ₃					
	η (Pa.s)	$\dot{\gamma}$ (sec ⁻¹)	τ (Pa)	$\log(\dot{\gamma})$	$\log(\tau)$	$\log(\eta)$
0.3	0.09	0.396	0.04	-0.40	-1.44	-1.04
0.6	0.09	0.792	0.07	-0.10	-1.15	-1.05
1.5	0.10	1.980	0.20	0.30	-0.69	-0.99
3.0	0.09	3.960	0.36	0.60	-0.45	-1.05
6.0	0.07	7.920	0.59	0.90	-0.23	-1.13
12.0	0.06	15.800	0.90	1.20	-0.05	-1.24
30.0	0.04	39.600	1.51	1.60	0.18	-1.42
60.0	0.03	79.200	2.20	1.90	0.34	-1.56
0.3	0.19	0.396	0.08	-0.40	-1.12	-0.72
Stop 5 min						
0.3	0.37	0.396	0.15	-0.40	-0.84	-0.43
rpm	Small sample DV II+ Spindle No.31					
	50% RC11 + 1 wt% Binder + 0.14 wt% Na ₂ SiO ₃					
	η (Pa.s)	$\dot{\gamma}$ (sec ⁻¹)	τ (Pa)	$\log(\dot{\gamma})$	$\log(\tau)$	$\log(\eta)$
0.3	3.50	0.102	0.36	-0.99	-0.45	0.54
0.6	4.30	0.204	0.88	-0.69	-0.06	0.63
1.5	2.90	0.510	1.48	-0.29	0.17	0.46
3.0	1.96	1.020	2.00	0.01	0.30	0.29
6.0	1.24	2.040	2.52	0.31	0.40	0.09
12.0	0.76	4.080	3.11	0.61	0.49	-0.12
30.0	0.39	10.200	3.95	1.01	0.60	-0.41
60.0	0.23	20.400	4.78	1.31	0.68	-0.63
0.3	7.10	0.102	0.72	-0.99	-0.14	0.85
Stop 5 min						
0.3	14.90	0.102	1.52	-0.99	0.18	1.17

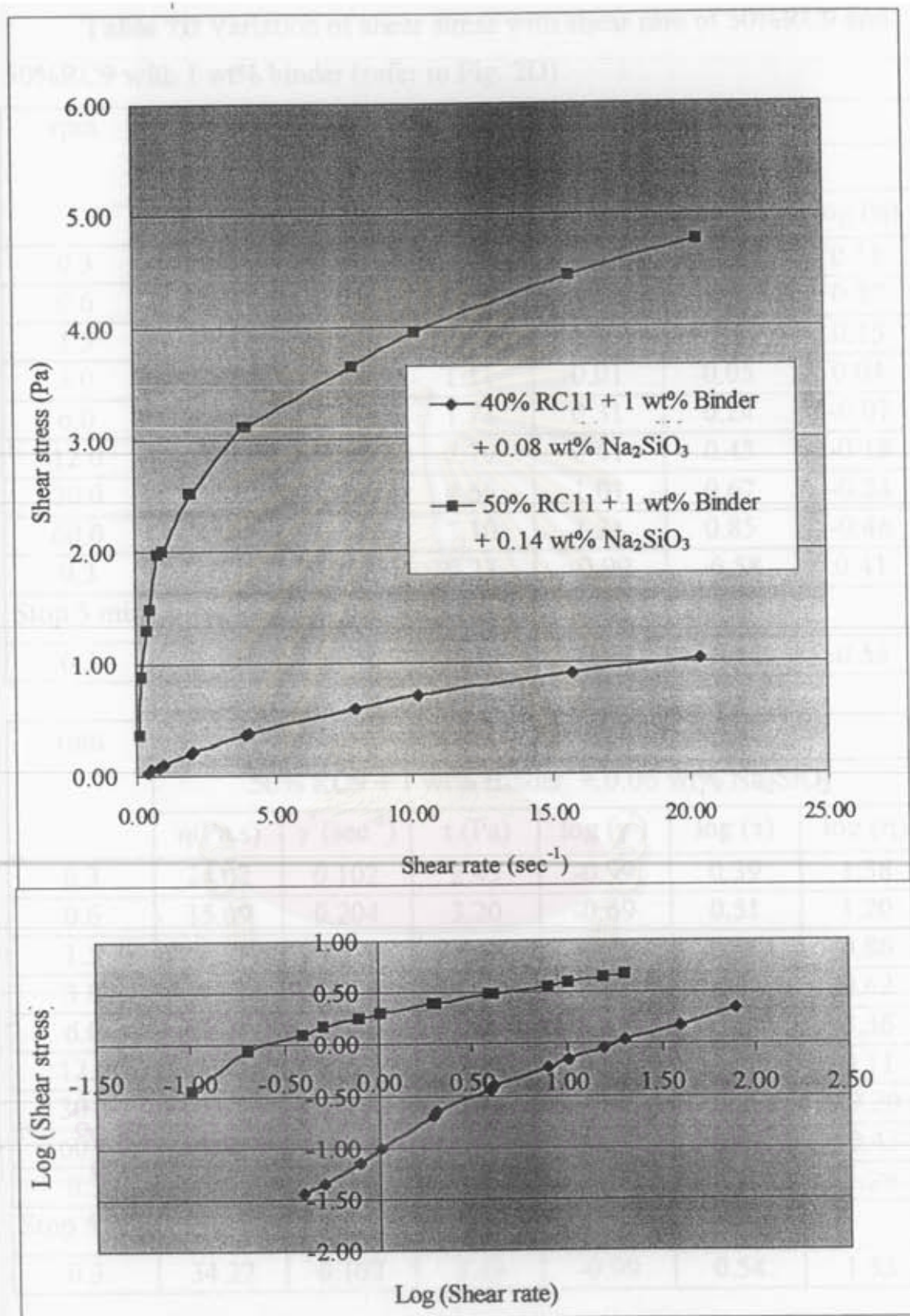


Fig. 1D Variation of shear stress with shear rate of 40%RC11 with 1 wt% binder and 50%RC11 with 1 wt% binder (Rechecking before spray drying and relating to Table 6D)

Table 7D Variation of shear stress with shear rate of 50%RC9 and 50%RC9 with 1 wt% binder (refer to Fig. 2D)

rpm	Small sample DV II+ Spindle No.31					
	50% RC9 + 0.10 wt% Na ₂ SiO ₃					
	η (Pa.s)	$\dot{\gamma}$ (sec ⁻¹)	τ (Pa)	$\log(\dot{\gamma})$	$\log(\tau)$	$\log(\eta)$
0.3	1.50	0.102	0.15	-0.99	-0.82	0.18
0.6	1.65	0.204	0.34	-0.69	-0.47	0.22
1.5	1.40	0.510	0.71	-0.29	-0.15	0.15
3.0	1.09	1.020	1.11	0.01	0.05	0.04
6.0	0.85	2.040	1.73	0.31	0.24	-0.07
12.0	0.66	4.080	2.70	0.61	0.43	-0.18
30.0	0.46	10.200	4.66	1.01	0.67	-0.34
60.0	0.35	20.400	7.10	1.31	0.85	-0.46
0.3	2.60	0.102	0.27	-0.99	-0.58	0.41
Stop 5 min						
0.3	3.40	0.102	0.35	-0.99	-0.46	0.53
rpm	Small sample DV II+ Spindle No.31					
	50% RC9 + 1 wt% Binder + 0.06 wt% Na ₂ SiO ₃					
	η (Pa.s)	$\dot{\gamma}$ (sec ⁻¹)	τ (Pa)	$\log(\dot{\gamma})$	$\log(\tau)$	$\log(\eta)$
0.3	24.02	0.102	2.45	-0.99	0.39	1.38
0.6	15.69	0.204	3.20	-0.69	0.51	1.20
1.5	7.20	0.510	3.67	-0.29	0.56	0.86
3.0	4.13	1.020	4.21	0.01	0.62	0.62
6.0	2.29	2.040	4.68	0.31	0.67	0.36
12.0	1.30	4.080	5.30	0.61	0.72	0.11
30.0	0.63	10.200	6.46	1.01	0.81	-0.20
60.0	0.38	20.400	7.66	1.31	0.88	-0.43
0.3	19.61	0.102	2.00	-0.99	0.30	1.29
Stop 5 min						
0.3	34.22	0.102	3.49	-0.99	0.54	1.53

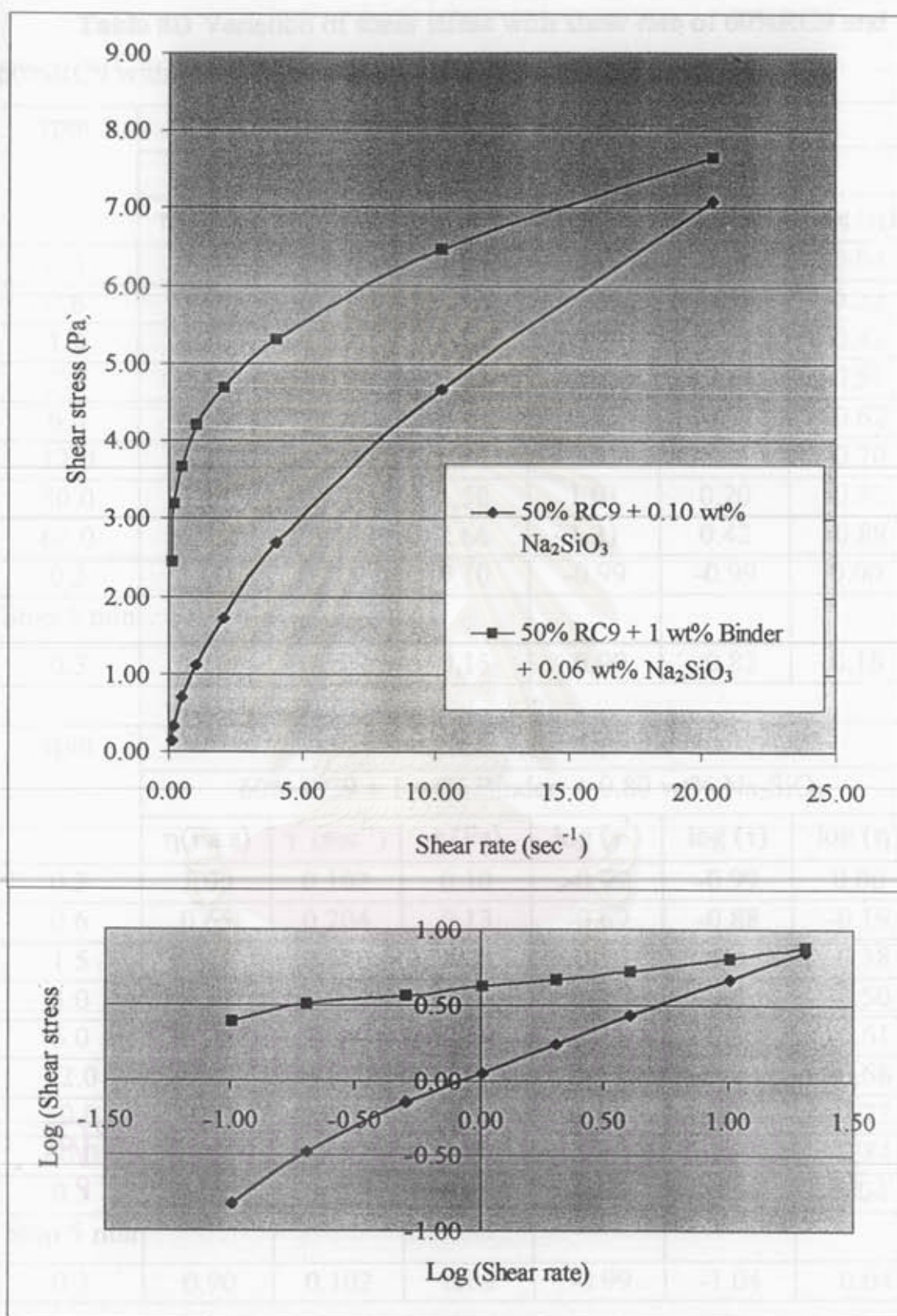


Fig. 2D Variation of shear stress with shear rate of 50%RC9 with and without 1wt%binder
(Rechecking before spray drying and relating to Table 7D)

Table 8D Variation of shear stress with shear rate of 60%RC9 and 60%RC9 with 1 wt% binder (refer to Fig. 3D)

rpm	Small sample DV II+ Spindle No.31					
	60% RC9 + 0.55 wt% Na ₂ SiO ₃					
	η (Pa.s)	$\dot{\gamma}$ (sec ⁻¹)	τ (Pa)	$\log(\dot{\gamma})$	$\log(\tau)$	$\log(\eta)$
0.3	0.90	0.102	0.09	-0.99	-1.04	-0.04
0.6	0.60	0.204	0.12	-0.69	-0.91	-0.22
1.5	0.38	0.510	0.19	-0.29	-0.71	-0.42
3.0	0.31	1.020	0.32	0.01	-0.50	-0.51
6.0	0.24	2.040	0.49	0.31	-0.31	-0.62
12.0	0.20	4.080	0.81	0.61	-0.09	-0.70
30.0	0.15	10.200	1.58	1.01	0.20	-0.81
60.0	0.13	20.400	2.66	1.31	0.42	-0.88
0.3	1.00	0.102	0.10	-0.99	-0.99	0.00
Stop 5 min						
0.3	1.50	0.102	0.15	-0.99	-0.82	0.18
rpm	Small sample DV II+ Spindle No.31					
	60% RC9 + 1 wt% Binder + 0.80 wt% Na ₂ SiO ₃					
	η (Pa.s)	$\dot{\gamma}$ (sec ⁻¹)	τ (Pa)	$\log(\dot{\gamma})$	$\log(\tau)$	$\log(\eta)$
0.3	1.00	0.102	0.10	-0.99	-0.99	0.00
0.6	0.65	0.204	0.13	-0.69	-0.88	-0.19
1.5	0.42	0.510	0.21	-0.29	-0.67	-0.38
3.0	0.32	1.020	0.33	0.01	-0.49	-0.50
6.0	0.25	2.040	0.50	0.31	-0.30	-0.61
12.0	0.21	4.080	0.85	0.61	-0.07	-0.68
30.0	0.17	10.200	1.72	1.01	0.24	-0.77
60.0	0.15	20.400	2.98	1.31	0.47	-0.84
0.3	1.10	0.102	0.11	-0.99	-0.95	0.04
Stop 5 min						
0.3	0.90	0.102	0.09	-0.99	-1.04	-0.04

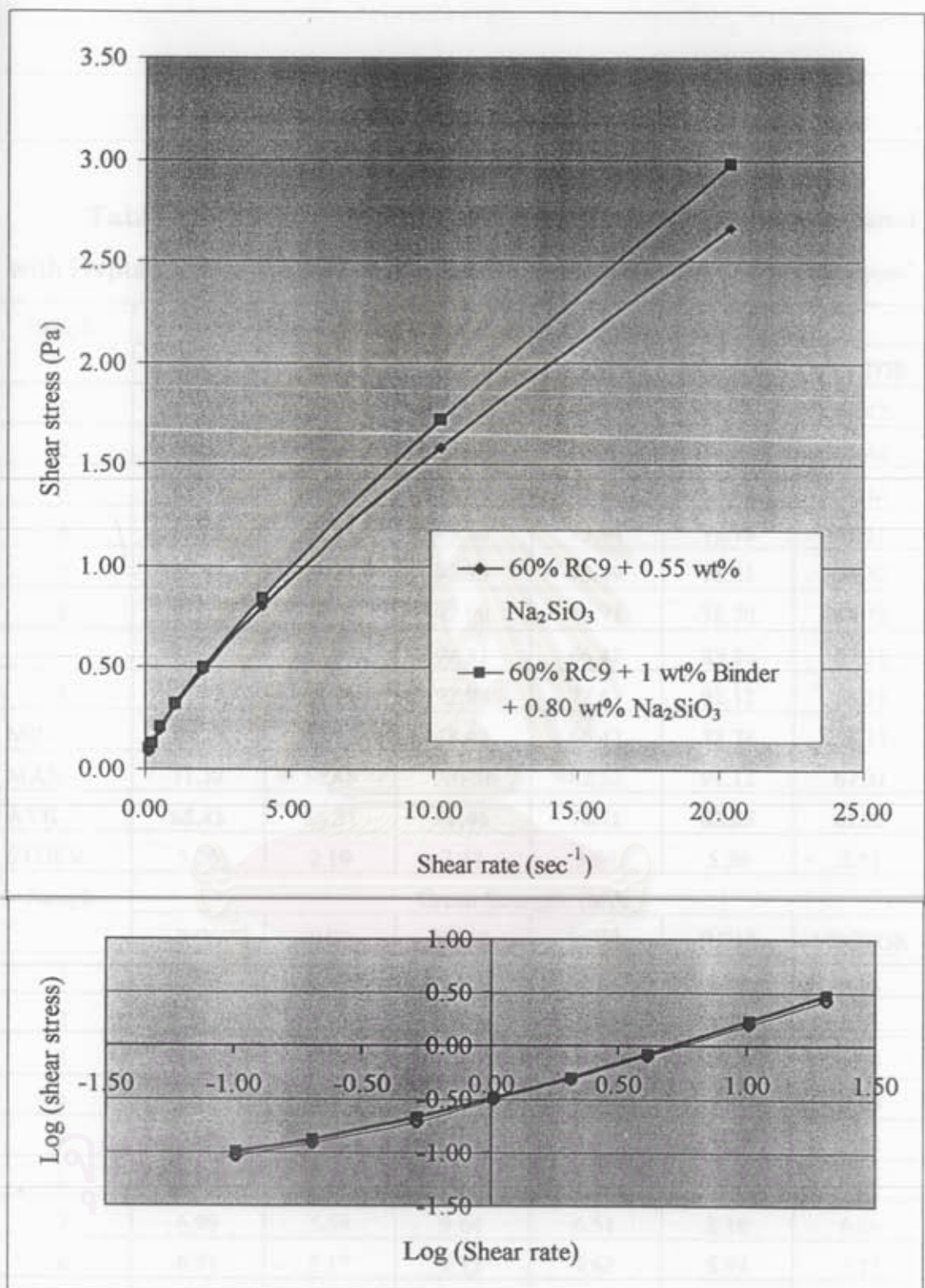


Fig. 3D Variation of shear stress with shear rate of 60%RC9 with and without 1wt%binder

(Rechecking before spray drying and relating to Table 8D)

APPENDIX E

Modulus of rupture data (Green strength)

Table 1E Green strength of RC slips at 50 wt% solid compared with Hypure Vector®. (refer to Fig. 5.13) $1\text{MPa} = 1\text{MN/m}^2 = 10.019716\text{ kg/cm}^2$

Sample	Green strength (kg/cm ²)					
	RC6	RC9	RC10	RC11	RC12	VECTOR
1	60.08	55.15	82.34	78.70	81.16	66.42
2	59.31	54.37	100.36	66.76	86.52	66.42
3	67.82	58.45	78.42	82.52	83.62	62.13
4	65.03	54.11	93.54	73.44	73.78	61.11
5	60.08	53.14	92.73	72.49	78.21	54.37
6	71.32	58.03	92.99	79.71	78.70	64.72
7	71.32	56.95	98.31	66.42	83.26	67.31
8	68.43	52.75	92.99	77.67	91.12	53.14
MIN	59.31	52.75	78.42	66.42	73.78	53.14
MAX	71.32	58.45	100.36	82.52	91.12	67.31
AVG.	65.42	55.37	91.46	74.71	82.05	61.95
STDEV.	5.06	2.19	7.47	5.96	5.36	5.51
Sample	Green Strength (MPa)					
	RC6	RC9	RC10	RC11	RC12	VECTOR
1	5.89	5.41	8.07	7.72	7.96	6.51
2	5.82	5.33	9.84	6.55	8.49	6.51
3	6.65	5.73	7.69	8.09	8.20	6.09
4	6.38	5.31	9.17	7.20	7.24	5.99
5	5.89	5.21	9.09	7.11	7.67	5.33
6	6.99	5.69	9.12	7.82	7.72	6.35
7	6.99	5.59	9.64	6.51	8.16	6.60
8	6.71	5.17	9.12	7.62	8.94	5.21
MIN	5.82	5.17	7.69	6.51	7.24	5.21
MAX	6.99	5.73	9.84	8.09	8.94	6.60
AVG.	6.42	5.43	8.97	7.33	8.05	6.08
STDEV.	0.50	0.21	0.73	0.58	0.53	0.54

Table 2E Modulus of rupture (green strength) of RC9 slips before and after spray drying compared with Hypure Vector®.
(refer to Fig. 5.25) $1\text{MPa} = 1\text{MN/m}^2 = 10.019716 \text{ kg/cm}^2$

Sample	Green strength before spray drying (MPa)			
	50RC9	50RC9+1B	60RC9	60RC9+1B
1	5.06	5.97	6.82	6.95
2	5.46	5.97	6.04	7.37
3	5.59	6.12	5.97	8.15
4	5.41	5.97	6.74	7.90
5	5.59	5.88	6.65	7.16
6	6.03	5.74	6.55	6.90
7	5.24	5.41	6.77	7.51
8		6.12	6.42	7.71
MIN	5.06	5.41	5.97	6.90
MAX	6.03	6.12	6.82	8.15
AVG.	5.48	5.89	6.49	7.46
STDEV.	0.31	0.23	0.33	0.45
Sample	Green strength after spray drying (MPa)			
	50RC9	50RC9+B	60RC9	VECTOR
1	4.70	4.95	6.68	6.51
2	4.45	6.24	6.25	6.51
3	4.36	4.67	6.19	6.09
4	5.44	4.95	5.84	5.99
5	5.37	6.29	6.27	5.33
6	4.95	5.94	5.44	6.35
7	5.14	5.40	5.33	6.60
8	4.70	5.55	6.27	5.21
MIN	4.36	4.67	5.33	5.21
MAX	5.44	6.29	6.68	6.60
AVG.	4.89	5.50	6.03	6.08
STDEV.	0.40	0.62	0.46	0.54

APPENDIX F

Granule properties

Table 1F Granule size distribution of samples by sieve analysis.
(refer to Fig. 5.27)

Samples (fixed condition)	Weight percent over than (%)						
	VECTOR	40RC11	50RC11	50RC9	50RC9	60RC9	60RC9
		+0.08Na	+0.14Na	+0.1Na	+0.06Na	+0.55Na	+0.08Na
		+1B	+1B		+1B		+1B
+180 μm	77.80	0.36	0.60	0.56	0.56	1.84	4.24
+125 μm	90.40	0.56	1.12	1.04	0.92	3.16	6.16
+106 μm	93.20	0.72	1.92	1.96	1.24	3.96	7.16
+75 μm	98.00	3.40	8.56	13.76	3.12	7.84	9.04
+45 μm	99.60	69.40	76.56	80.38	78.36	82.80	76.62
-45 μm	99.76	99.56	99.68	99.28	99.56	99.44	99.85
Samples (varied Condition)	Weight percent over than (%)						
	50RC11	46RC11	46RC11	50RC11	50RC11		
	+0.28Na		+0.1Na	+0.3Na	+0.3Na		
			+1B	+1B	+2B		
+180 μm	1.04	1.16	0.89	0.72	0.96		
+125 μm	1.56	1.56	1.56	1.12	1.56		
+106 μm	1.84	1.84	1.91	1.44	2.12		
+75 μm	3.48	15.40	17.38	3.16	4.84		
+45 μm	81.08	92.28	79.96	75.56	75.92		
-45 μm	99.80	99.88	99.87	99.56	99.52		

Table 2F Granule size distribution of samples, measured by stereomicroscope. (refer to Fig. 5.28-5.29)

Samples (fixed condition)	Percent finer than (%)						
	VECTOR	40RC11	50RC11	50RC9	50RC9	60RC9	60RC9
		+0.08Na	+0.14Na	+0.1Na	+0.06Na	+0.55Na	+0.08Na
	+1B	+1B		+1B		+1B	
-10 μm	0.00	7.25	10.10	5.10	17.72	10.13	5.26
-20 μm	0.00	34.78	54.55	48.98	46.84	46.84	41.05
-30 μm	0.00	73.91	78.79	77.55	64.56	64.56	72.63
-40 μm	0.00	88.41	91.92	94.90	70.89	78.48	88.42
-50 μm	0.00	94.20	95.96	97.96	93.67	94.94	98.95
-56 μm	3.23	94.20	95.96	97.96	93.67	94.94	98.95
-60 μm	3.23	95.65	98.99	100.00	97.47	97.47	100.00
-70 μm	3.23	97.10	100.00	100.00	100.00	97.47	100.00
-80 μm	3.23	98.55	100.00	100.00	100.00	98.73	100.00
-90 μm	3.23	100.00	100.00	100.00	100.00	98.73	100.00
-100 μm	3.23	100.00	100.00	100.00	100.00	98.73	100.00
-110 μm	3.23	100.00	100.00	100.00	100.00	98.73	100.00
-111 μm	12.90	100.00	100.00	100.00	100.00	98.73	100.00
-120 μm	12.90	100.00	100.00	100.00	100.00	100.00	100.00
-168 μm	22.58	100.00	100.00	100.00	100.00	100.00	100.00
-222 μm	41.94	100.00	100.00	100.00	100.00	100.00	100.00
-278 μm	70.97	100.00	100.00	100.00	100.00	100.00	100.00
-333 μm	80.65	100.00	100.00	100.00	100.00	100.00	100.00
-389 μm	90.32	100.00	100.00	100.00	100.00	100.00	100.00
-444 μm	96.77	100.00	100.00	100.00	100.00	100.00	100.00
-667 μm	100.00	100.00	100.00	100.00	100.00	100.00	100.00
Samples (varied Condition)	Percent finer than (%)						
	50RC11	46RC11	46RC11	50RC11	50RC11		
	+0.28Na		+0.1Na	+0.3Na	+0.3Na		
			+1B	+1B	+2B		
-10 μm	0.00	0.00	10.96	8.06	11.11		
-20 μm	15.00	10.81	47.95	38.71	26.39		
-30 μm	51.67	29.73	82.19	70.97	69.44		
-40 μm	75.00	48.65	93.15	85.48	86.11		
-50 μm	91.67	86.49	97.26	90.32	94.44		
-56 μm	91.67	86.49	97.26	90.32	94.44		
-60 μm	100.00	94.59	100.00	96.77	95.83		
-70 μm	100.00	97.30	100.00	96.77	97.22		
-80 μm	100.00	97.30	100.00	96.77	98.61		
-90 μm	100.00	97.30	100.00	98.39	98.61		
-100 μm	100.00	100.00	100.00	100.00	100.00		

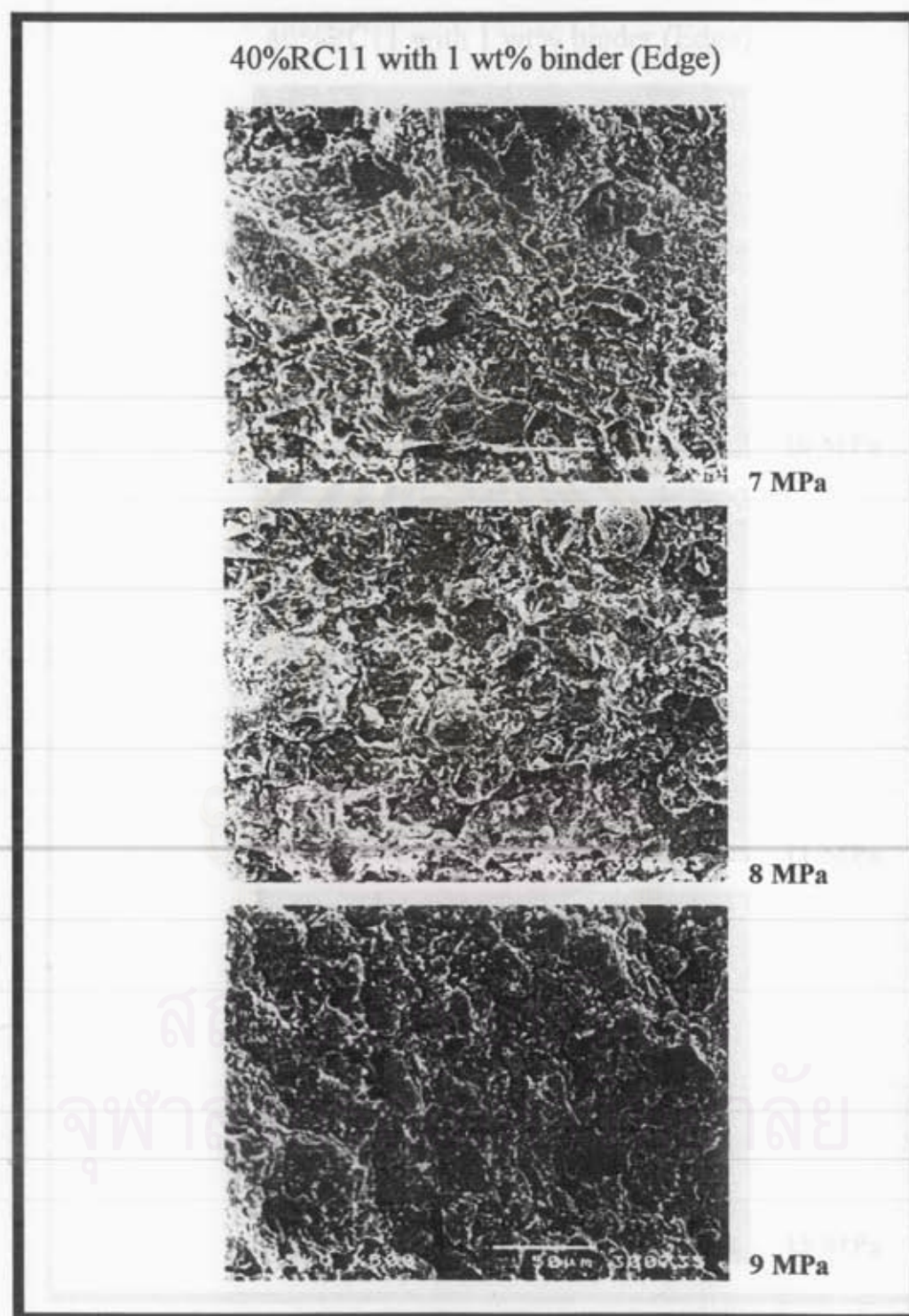


Fig. 1F Granule deformation of 40%RC11 with 1 wt% binder at various pressures – 7 to 9 MPa. (Fracture surface by SEM x 500)

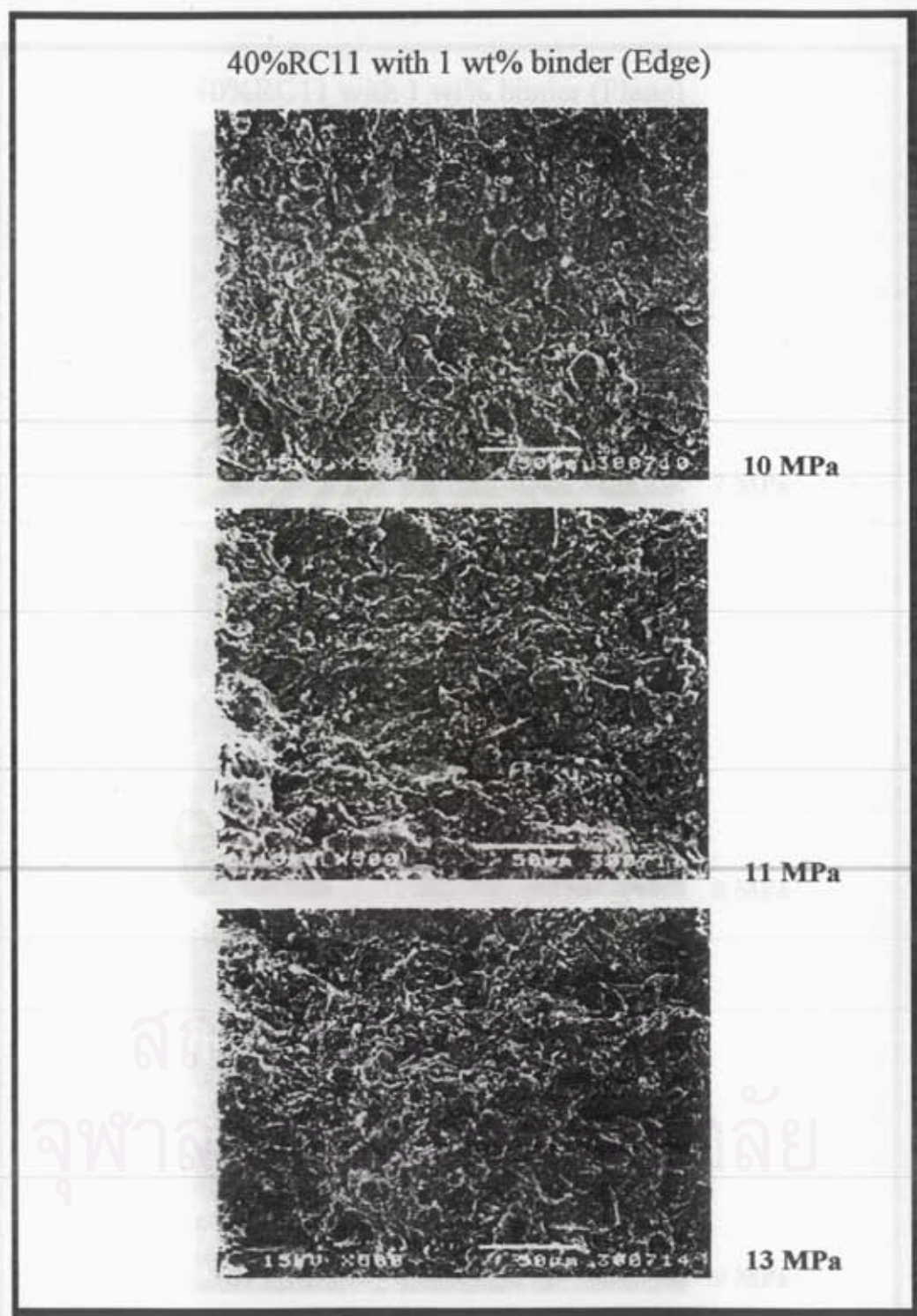


Fig. 2F Granule deformation of 40%RC11 with 1 wt% binder at various pressures – 10 to 13 MPa. (Fracture surface by SEM x 500)

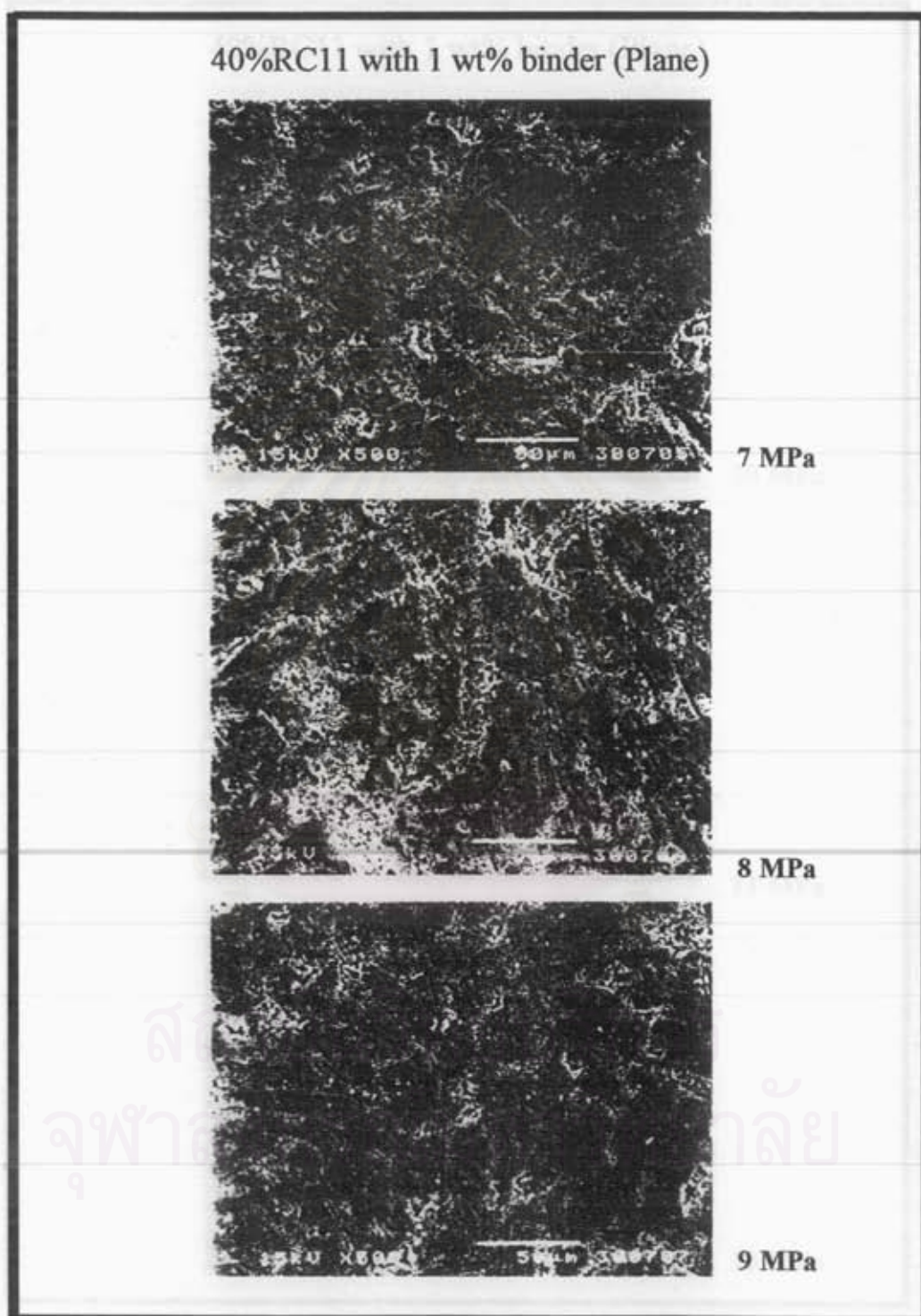


Fig. 3F Surface of 40%RC11 with 1wt% binder at various pressures – 7 to 9 MPa. (Fracture surface by SEM x 500)

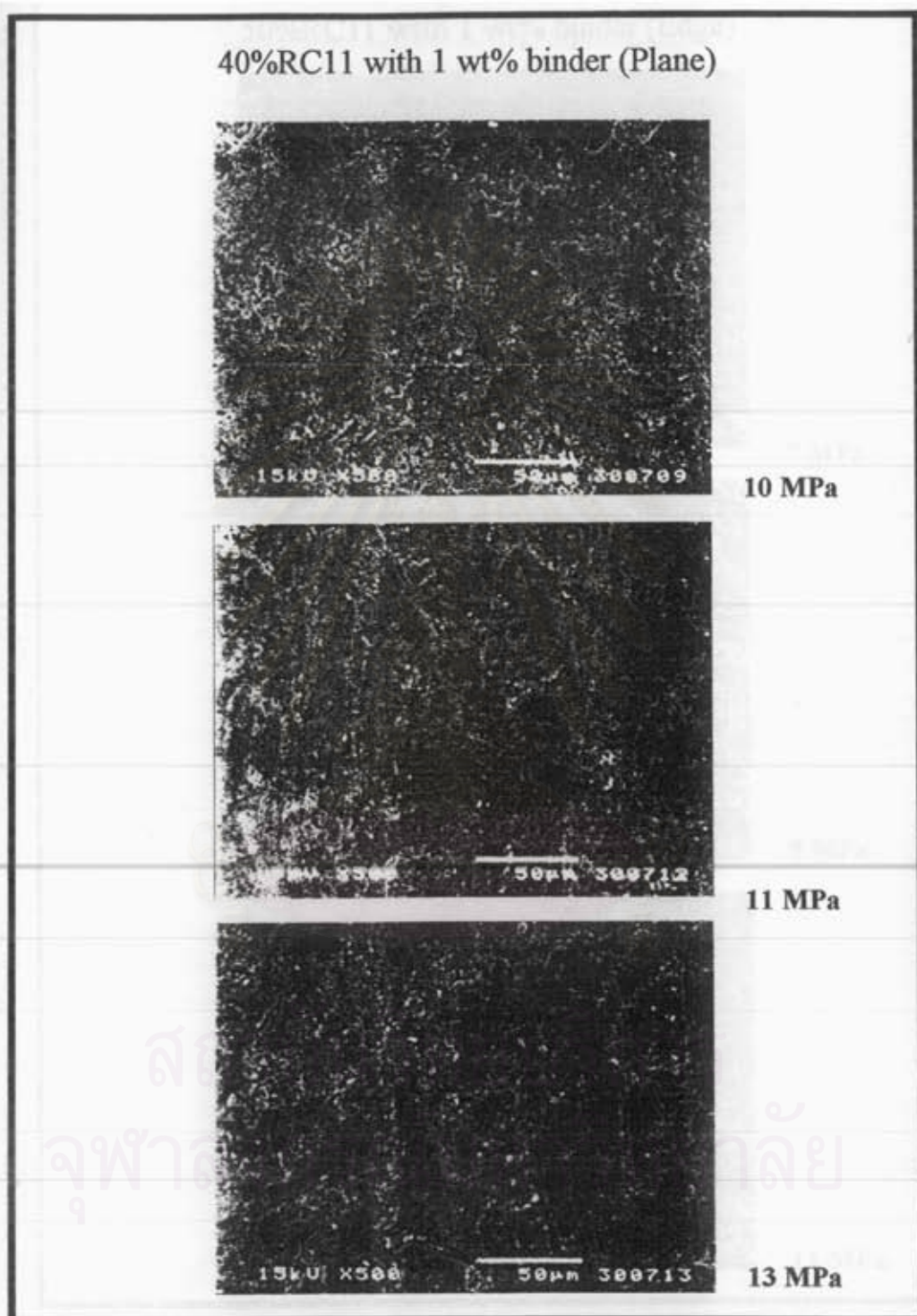


Fig. 4F Surface of 40%RC11 with 1wt% binder at various pressures – 10 to 13 MPa. (Fracture surface by SEM x 500)

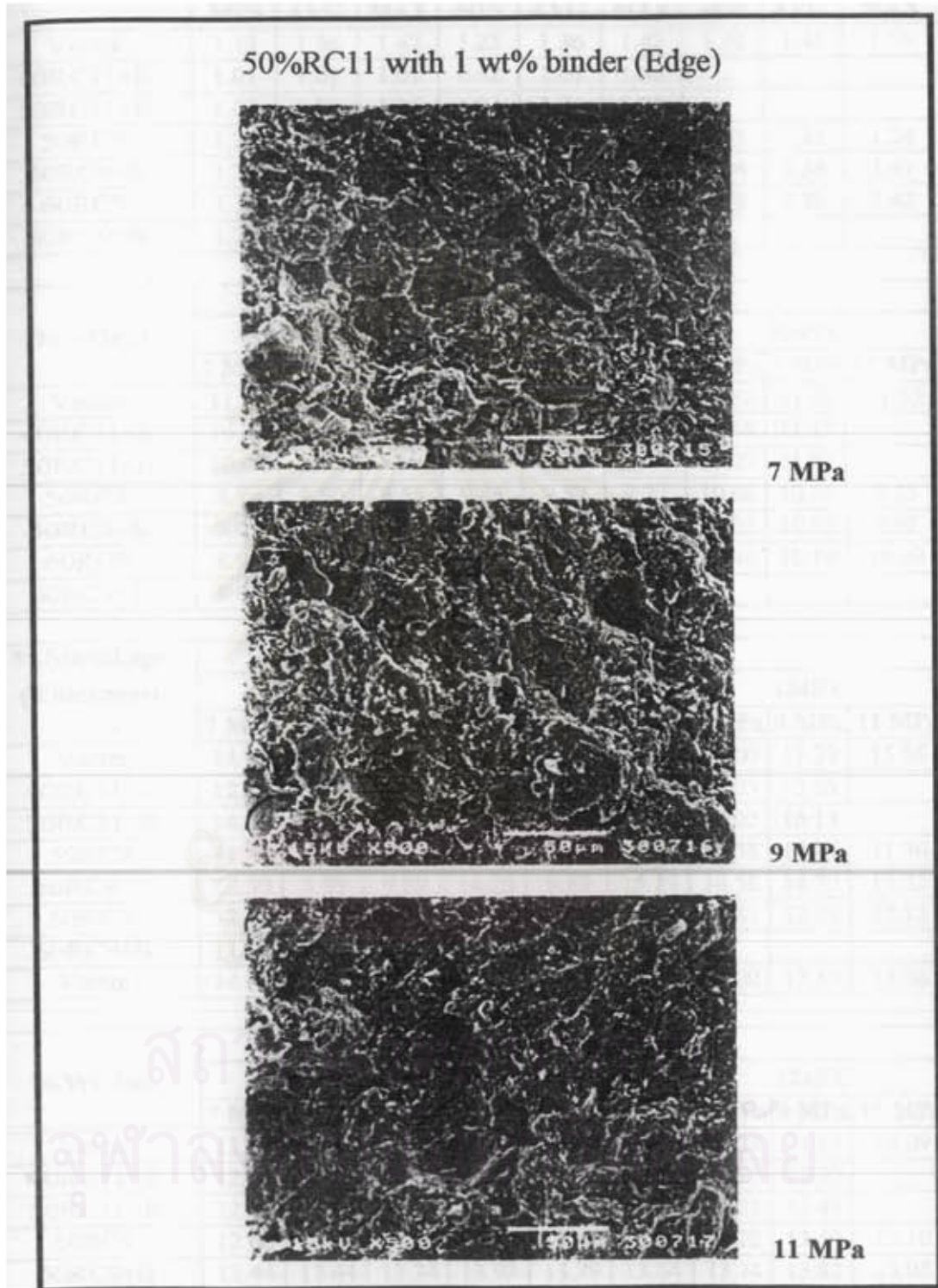


Fig. 5F Granule deformation of 50%RC11 with 1 wt% binder at various pressures. (Fracture surface by SEM x 500)

Table 3F %Shrinkage and %wt. loss of pressed samples (related to Fig. 5.43 to 5.44)¹⁵⁷

Mould-dry	%Shrinkage								
	7 MPa (70 Bar)			9 MPa (90 Bar)			11 MPa (110 Bar)		
	MIN	AVG.	MAX	MIN	AVG.	MAX	MIN	AVG.	MAX
Vector	1.13	1.36	1.47	1.23	1.36	1.42	1.32	1.48	1.56
40RC11+B	1.01	1.01	1.01	0.92	1.01	1.06			
50RC11+B	1.01	1.08	1.15	1.24	1.26	1.28			
50RC9	1.23	1.25	1.28	1.24	1.24	1.24	1.15	1.21	1.24
50RC9+B	1.24	1.32	1.47	1.15	1.24	1.33	1.24	1.36	1.47
60RC9	1.15	1.27	1.33	1.38	1.44	1.47	1.33	1.36	1.42
60RC9+B	1.38	1.47	1.56	1.22	1.28	1.33			

Dry-Fired	%Shrinkage								
	1150°C			1200°C			1245°C		
	7 MPa	9 MPa	11 MPa	7 MPa	9 MPa	11 MPa	7 MPa	9 MPa	11 MPa
Vector	11.04	10.72	10.26	11.51	11.14	10.68	11.82	11.21	11.62
40RC11+B	10.43	10.11		10.97	10.76		11.58	11.17	
50RC11+B	10.04	9.85		10.51	10.44		11.29	11.01	
50RC9	8.74	8.50	8.55	9.85	9.57	9.27	10.68	10.36	9.25
50RC9+B	9.01	8.83	8.74	10.08	9.90	9.95	11.05	10.63	9.92
60RC9	8.94	8.79	8.69	9.72	9.40	9.23	10.46	10.19	10.09
60RC9+B	8.66	8.67		9.41	9.23				

%Shrinkage (Thickness)	%Shrinkage								
	1150°C			1200°C			1245°C		
	7 MPa	9 MPa	11 MPa	7 MPa	9 MPa	11 MPa	7 MPa	9 MPa	11 MPa
Vector	14.89	15.22	13.33	14.89	15.22	15.22	20.00	17.39	15.56
40RC11+B	12.77	12.09		15.63	13.98		15.05	13.55	
50RC11+B	14.58	13.04		15.72	14.02		17.02	16.13	
50RC9	11.46	11.11	13.04	12.50	11.11	13.04	14.58	14.13	11.36
50RC9+B	12.50	8.89	9.89	14.29	9.89	15.79	14.58	14.29	13.33
60RC9	12.50	7.87	11.11	13.54	11.11	11.11	13.83	12.22	12.22
60RC9+B	11.11	10.34		12.22	11.93				
Vector	14.89	15.22	13.33	14.89	15.22	15.22	20.00	17.39	15.56

%Wt. loss	%Wt. loss								
	1150°C			1200°C			1245°C		
	7 MPa	9 MPa	11 MPa	7 MPa	9 MPa	11 MPa	7 MPa	9 MPa	11 MPa
Vector	13.74	13.46	13.64	14.37	14.22	14.26	14.37	14.17	14.09
40RC11+B	12.46	12.66		13.52	13.77		13.51	13.30	
50RC11+B	12.51	12.75		13.07	13.15		13.21	13.45	
50RC9	12.66	12.79	12.89	13.32	13.43	13.68	13.22	13.49	13.10
50RC9+B	13.44	13.44	13.34	13.93	13.79	13.64	13.74	13.82	13.95
60RC9	12.79	12.73	12.74	13.39	13.25	13.33	13.27	13.29	13.33
60RC9+B	13.30	13.25		13.82	13.89				

Table 4F Green density of pressed samples (related to Fig. 5.42)

Green density	Green density (g/cm ³)								
	70 Bar (7 MPa)			90 Bar (9 MPa)			110 Bar (11 MPa)		
	MIN	AVG.	MAX	MIN	AVG.	MAX	MIN	AVG.	MAX
Vector	1.73	1.77	1.80	1.79	1.82	1.84	1.86	1.88	1.89
40RC11+B	1.76	1.76	1.77	1.79	1.80	1.81			
50RC11+B	1.70	1.73	1.75	1.75	1.78	1.81			
50RC9	1.71	1.75	1.77	1.78	1.83	1.87	1.84	1.88	1.94
50RC9+B	1.73	1.74	1.76	1.82	1.85	1.87	1.76	1.84	1.90
60RC9	1.73	1.76	1.78	1.86	1.88	1.90	1.76	1.84	1.90
60RC9+B	1.85	1.86	1.86	1.91	1.91	1.91			

Table 5F Bulk density, %water absorption, %apparent porosity and specific gravity of pressed samples (related to Fig. 5.45 and Fig. 6F)

B=D/(M-S)	Bulk density (g/cm ³)								
	1150°C			1200°C			1245°C		
	7 MPa	9 MPa	11 MPa	7 MPa	9 MPa	11 MPa	7 MPa	9 MPa	11 MPa
Vector	2.40	2.45	2.43	2.43	2.49	2.48	2.47	2.48	2.47
40RC11+B	2.28	2.34	2.32	2.36	2.31	2.41	2.38	2.42	2.42
50RC9	2.17	2.22	2.33	2.29	2.29	2.34	2.29	2.39	2.44
50RC9+B	2.19	2.21	2.23	2.24	2.32	2.32	2.30	2.35	2.46
60RC9	2.18	2.23	2.29	2.25	2.34	2.39	2.37	2.44	2.48

A=(M-D)/D x 100	%Water absorption								
	1150°C			1200°C			1245°C		
	7 MPa	9 MPa	11 MPa	7 MPa	9 MPa	11 MPa	7 MPa	9 MPa	11 MPa
Vector	0.14	0.04	0.34	0.12	0.07	0.05	0.06	0.05	0.07
40RC11+B	4.84	4.11	3.84	2.9	3.58	2.05	2.36	1.36	1.64
50RC9	8.39	7.04	4.91	5.25	5.58	4.34	5.01	3.43	2.33
50RC9+B	7.84	7.59	6.58	6.37	4.34	4.85	4.98	3.81	2.58
60RC9	8.24	7.08	5.39	6.21	4.14	3.38	3.82	2.21	1.57

P=(M-D)/(M-S) x 100	%Apparent porosity								
	1150°C			1200°C			1245°C		
	7 MPa	9 MPa	11 MPa	7 MPa	9 MPa	11 MPa	7 MPa	9 MPa	11 MPa
Vector	0.32	0.10	0.81	0.29	0.17	0.13	0.16	0.13	0.18
40RC11+B	11.02	9.61	8.88	6.83	8.38	4.93	5.62	3.29	3.97
50RC9	18.24	15.63	11.44	12.02	12.78	10.13	11.47	8.78	5.68
50RC9+B	17.15	16.69	14.69	14.25	10.04	11.24	11.45	8.94	6.30
60RC9	17.91	15.72	12.33	13.96	9.70	8.06	8.96	5.40	3.89

T=D/(D-S)	Apparent specific gravity (g/cm ³)								
	1150°C			1200°C			1245°C		
	7 MPa	9 MPa	11 MPa	7 MPa	9 MPa	11 MPa	7 MPa	9 MPa	11 MPa
Vector	2.41	2.45	2.45	2.44	2.49	2.48	2.48	2.48	2.47
40RC11+B	2.56	2.59	2.55	2.53	2.55	2.53	2.52	2.50	2.51
50RC9	2.66	2.63	2.63	2.60	2.63	2.60	2.59	2.60	2.59
50RC9+B	2.64	2.65	2.62	2.61	2.58	2.61	2.60	2.58	2.62
60RC9	2.66	2.64	2.62	2.61	2.59	2.60	2.60	2.58	2.58

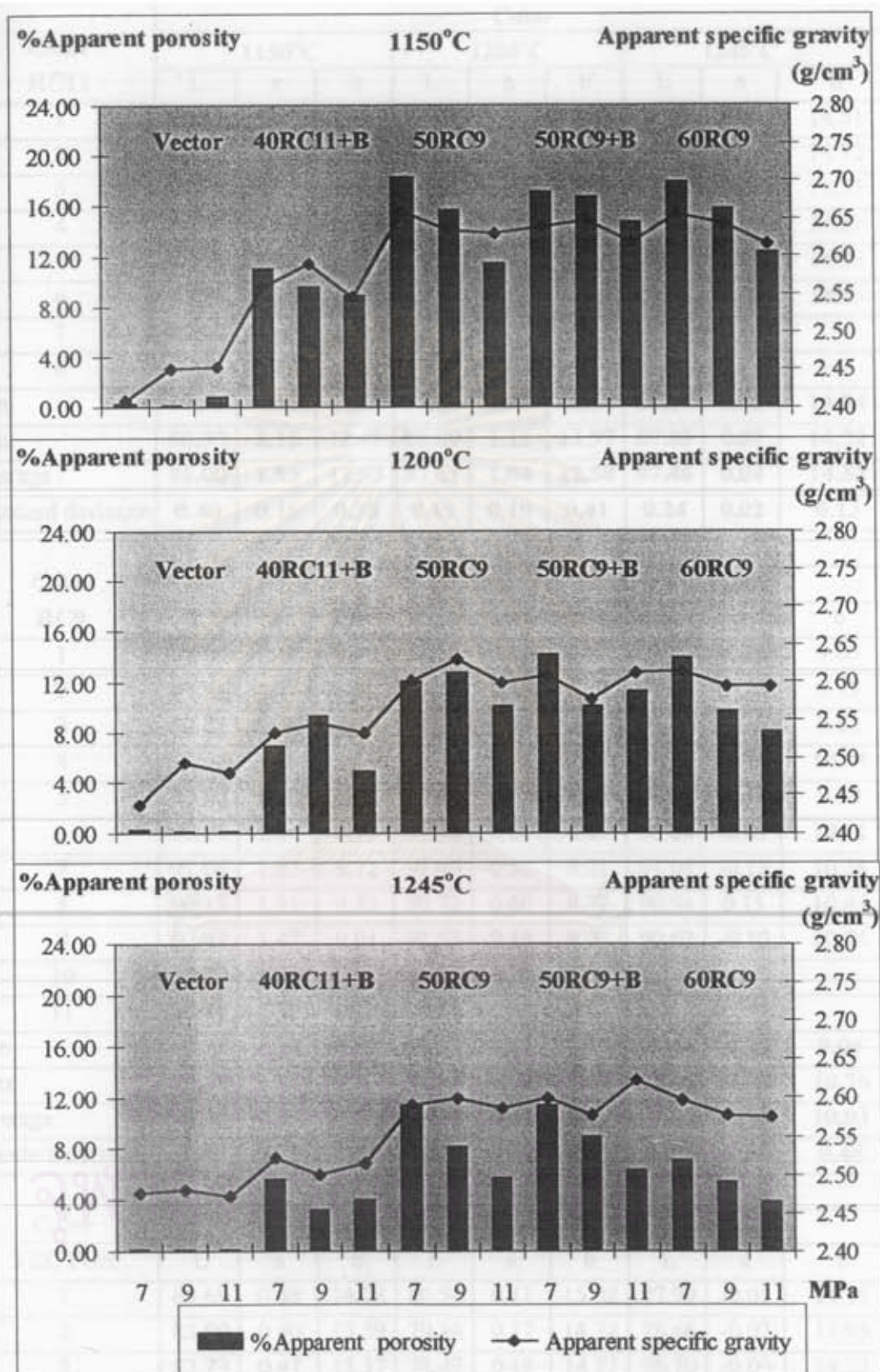


Fig. 6F Apparent specific gravity and apparent porosity of pressed samples at 1150°C-1245°C. (Table 5F)

Table 6F Color after firing of pressed samples

Color RC11	Color								
	1150°C			1200°C			1245°C		
	L	a	b	L	a	b	L	a	b
1	88.57	1.80	11.33	88.09	0.90	12.29	87.83	0.05	14.51
2	87.81	2.05	12.15	87.86	1.12	12.17	87.29	0.07	14.72
3	87.88	2.07	12.04	87.24	1.03	12.97	87.70	0.03	14.55
4	88.35	1.83	11.64	87.24	1.11	12.89	87.39	0.05	14.57
5	88.24	1.96	11.86				87.30	0.01	14.34
6	87.53	2.15	12.11				87.27	0.04	14.45
7	88.19	1.67	11.79						
8	87.45	1.89	12.48						
Min	87.45	1.67	11.33	87.24	0.90	12.17	87.27	0.01	14.34
Max	88.57	2.15	12.48	88.09	1.12	12.97	87.83	0.07	14.72
Average	88.00	1.93	11.93	87.61	1.04	12.58	87.46	0.04	14.52
Standard deviation	0.40	0.16	0.35	0.43	0.10	0.41	0.24	0.02	0.13
Color RC9	Color								
	1150°C			1200°C			1245°C		
	L	a	b	L	a	b	L	a	b
1	91.68	1.17	8.18	91.63	0.32	8.30	92.40	-0.29	9.05
2	91.41	1.27	8.33	90.65	0.48	8.77	91.51	-0.23	9.99
3	90.87	1.38	8.88	90.98	0.48	8.67	90.46	0.06	9.83
4	91.44	1.25	8.54	92.24	0.38	7.65	91.33	-0.16	10.04
5	90.90	1.40	8.90	91.68	0.38	8.30	91.67	-0.19	9.79
6	90.71	1.44	8.99	91.70	0.27	8.49	90.84	-0.07	10.11
7	91.06	1.22	8.72	91.40	0.52	8.21	91.05	-0.13	10.28
8	90.15	1.51	9.35	90.72	0.60	8.77	90.94	0.15	10.45
9	90.42	1.47	9.01	91.55	0.44	8.23	90.62	-0.10	10.76
10	90.82	1.38	8.90	91.93	0.36	8.04			
11	90.36	1.38	9.16	90.31	0.58	9.00			
Min	90.15	1.17	8.18	90.31	0.27	7.65	90.46	-0.29	9.05
Max	91.68	1.51	9.35	92.24	0.60	9.00	92.40	0.15	10.76
Average	90.89	1.35	8.81	91.34	0.44	8.40	91.20	-0.11	10.03
Standard deviation	0.48	0.11	0.35	0.60	0.11	0.39	0.60	0.14	0.48
Color VECTOR	Color								
	1150°C			1200°C			1245°C		
	L	a	b	L	a	b	L	a	b
1	85.69	0.58	14.28	80.50	0.11	15.02	77.99	-0.01	14.19
2	83.99	0.40	13.59	79.14	0.17	14.72	78.66	-0.07	13.95
3	82.73	0.47	13.37	78.47	0.18	14.77	76.70	-0.09	14.22
Min	82.73	0.40	13.37	78.47	0.11	14.72	76.70	-0.09	13.95
Max	85.69	0.58	14.28	80.50	0.18	15.02	78.66	-0.01	14.22
Average	84.14	0.48	13.75	79.37	0.15	14.84	77.78	-0.06	14.12
Standard deviation	1.49	0.09	0.47	1.03	0.04	0.16	1.00	0.04	0.15

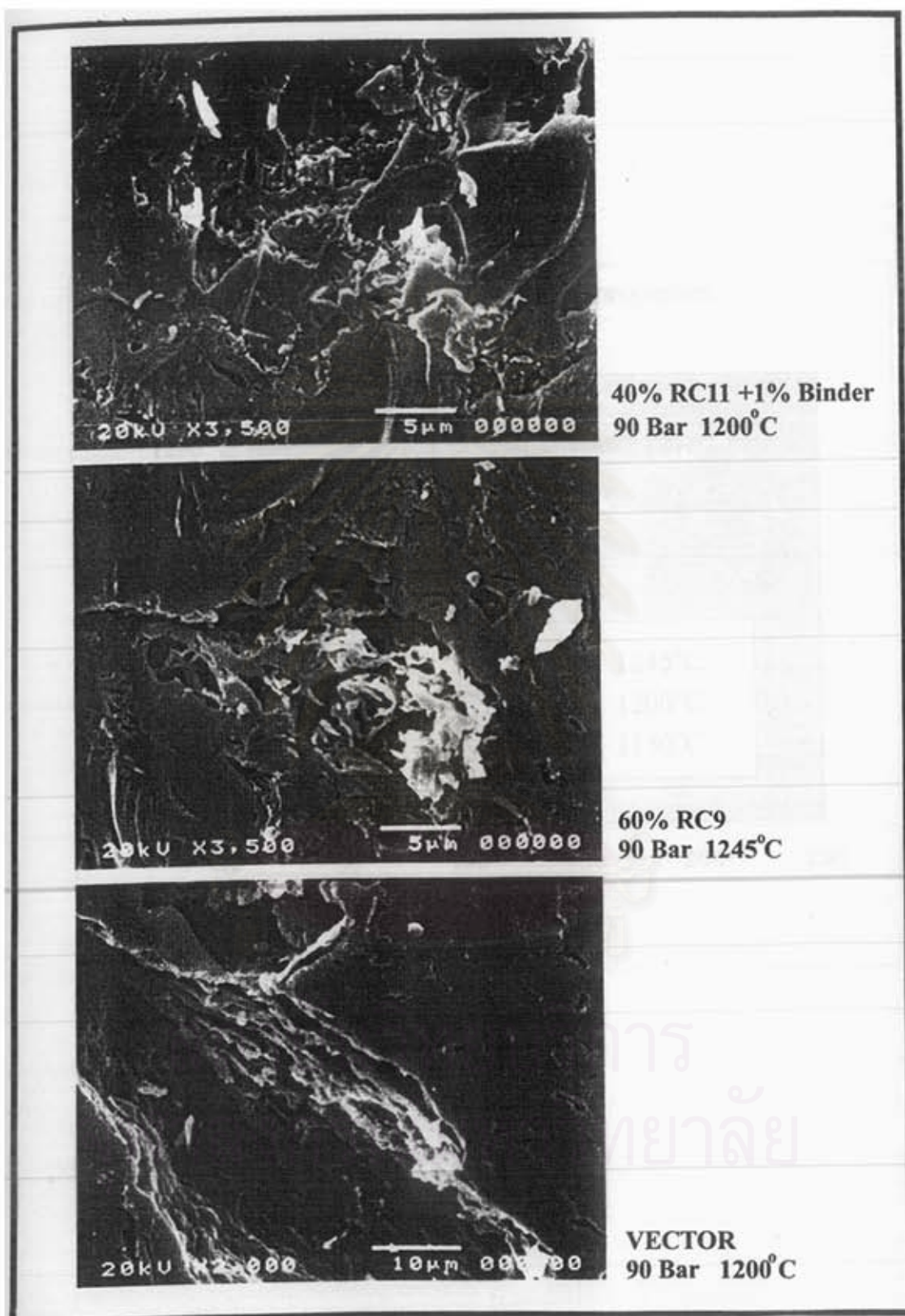
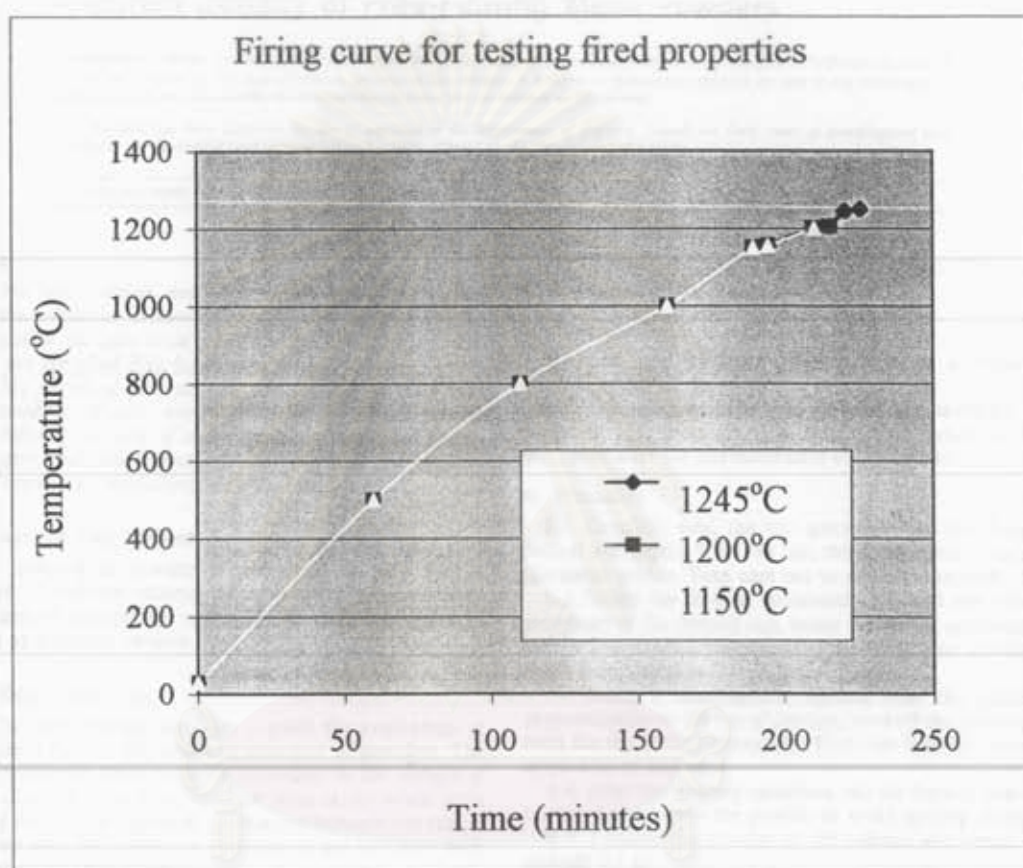


Fig. 8F Micrograph of pressed samples after firing.

APPENDIX G



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APPENDIX H

ASTM standard

Designation: B 212 - 89 (Reapproved 1995)¹

Standard Test Method for Apparent Density of Free-Flowing Metal Powders¹

This standard is issued under the fixed designation B 212; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

¹ NOTE—Keywords were added editorially in September 1995.

1. Scope

1.1 This test method describes a procedure for determining the apparent density of free-flowing metal powders and is suitable for only those powders that will flow unaided through the specified Hall flowmeter funnel.

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Summary of Test Method

2.1 A volume of powder is permitted to flow into a container of definite volume under controlled conditions. The weight of powder per unit volume is determined and reported as apparent density.

3. Significance and Use

3.1 This test method provides a guide for evaluation of the apparent density physical characteristic of powders. The density measured bears some relationship to the weight of powder that will fill a fixed volume press cavity when parts are being made. The degree of correlation between the results of this test and the quality of powders in use will vary with each particular application.

4. Apparatus

4.1 *Powder Flowmeter Funnel*²—A standard Hall flowmeter funnel (Fig. 1) having a calibrated orifice.

4.2 *Density Cup*²—A cylindrical cup (Fig. 1) having a capacity of $25 \pm 0.05 \text{ cm}^3$.

4.3 *Stand*²—A stand (Fig. 1) to support the powder flowmeter concentric with the density cup so that the bottom of the powder flowmeter orifice is 1 in. (25 mm) above the top of the density cup when the apparatus is assembled as shown in Fig. 1.

4.4 *Base*—A level, vibration-free base to support the powder flowmeter.

4.5 *Balance*, having a capacity of at least 200 g and a sensitivity of 0.1 g.

5. Test Specimen

5.1 The test specimen shall consist of a volume of approximately 30 to 40 cm^3 of metal powder.

5.2 The test specimen shall be tested as sampled. Note, however, that moisture, oils, stearic acid, stearates, waxes, etc., may alter the characteristics of the powder.

6. Procedure

6.1 Carefully load the test specimen into the flowmeter funnel and permit it to run into the density cup through the discharge orifice. Take care not to move the density cup.

6.2 When the powder completely fills and overflows the periphery of the density cup, rotate the funnel approximately 90° in a horizontal plane so that the remaining powder falls away from the cup.

6.3 Using a nonmagnetic spatula with the blade held perpendicular to the top of the cup, level off the powder flush with the top of the density cup. Take care to avoid jarring the apparatus at any time.

6.4 After the leveling operation, tap the density cup lightly on the side to settle the powder to avoid spilling in transfer.

6.5 Transfer the powder to the balance and weigh to the nearest 0.1 g.

7. Calculation

7.1 Calculate the apparent density as follows:

$$\text{Apparent density, g/cm}^3 = \text{weight in grams} \times 0.04$$

8. Report

8.1 Results shall be reported as apparent density to the nearest 0.01 g/cm^3 .

9. Precision and Bias

9.1 The following criteria should be used to judge acceptability of the results at the 95 % confidence level.

9.1.1 *Repeatability*—Duplicate results by the same operator should be considered suspect if they differ by more than 0.9 %.

9.1.2 *Reproducibility*—The results submitted by each of two laboratories should not be considered suspect unless they differ by more than 6.0 %.

10. Keywords

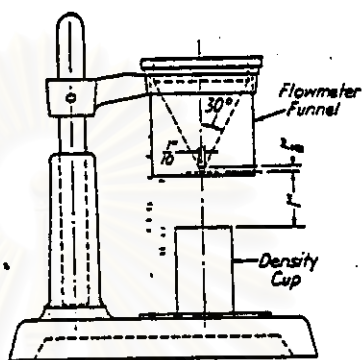
10.1 apparent density; Hall flowmeter funnel; metal powders

¹ This test method is under the jurisdiction of ASTM Committee B-9 on Metal Powders and Metal Powder Products and is the direct responsibility of Subcommittee B09.02 on Base Metal Powders.

Current edition approved Jan. 27, 1989. Published March 1989. Originally published as B 212 - 46 T. Last previous edition B 212 - 82.

² The flowmeter funnel, density cup, and stand are available from Alcan Powder and Pigments, Division of Alcan Aluminum Corp., 901 Lehigh Ave., Union, NJ 07083-7632.

B 212



Metric Equivalents

in.	mm
1/10	2.5
1/8	3.2
1	25

FIG. 1 Flowmeter Apparatus

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Designation: B 213 - 90

Standard Test Method for Flow Rate of Metal Powders¹

This standard is issued under the fixed designation B 213; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval. B 213

1. Scope

1.1 This test method covers the determination of the flow rate of metal powders and is suitable only for those powders which will flow unaided through the specified apparatus.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Significance and Use

2.1 The flow rate of a metal powder determines the rate of filling of a die cavity in the pressing of sintered metal parts or bearings. High flow rates (low flow times) are usually desired for high productivity. The test method may be part of the purchase agreement between powder manufacturer and P/M parts producers, or it may be an internal quality control test for powder blended by a parts producer. It is commonly applied to ferrous powders and copper base alloys, but may be used on other powders as well. The test is not applicable to wet or pasty mixtures of metal powders, since they will not flow through the funnel and are not commonly used in P/M processing.

3. Apparatus

3.1 *Powder Flowmeter Funnel*—A standard flowmeter funnel² (Fig. 1) having a calibrated orifice of 0.10 in. (2.54 mm) in diameter.

3.2 *Stand*²—A stand (Fig. 1) to support the powder flowmeter funnel.

3.3 *Base*—A level, vibration-free base to support the powder flowmeter.

3.4 *Stop Watch*.

3.5 *Balance*—A balance suitable for weighing accurately to 0.01 g.

4. Test Specimen

4.1 The test specimen shall be 50 g, weighed to the nearest 0.1 g.

5. Procedure

5.1 The test specimen shall be tested as sampled. It should be noted, however, that moisture, oils, stearic acid, stearates, waxes, etc., may alter the characteristics of the powder.

5.2 Carefully load the test specimen into the flowmeter funnel while keeping closed the discharge orifice at the bottom of the funnel by placing a dry finger under it. Take care that the short stem of the funnel is filled.

5.3 Start the stop watch simultaneously with removal of the finger from the discharge orifice and stop it at the instant the last of the powder leaves the funnel. Record the elapsed time in seconds.

6. Report

6.1 The elapsed time shall be multiplied by the correction factor (see Note) and the result reported in seconds to the nearest second.

NOTE —The manufacturer supplies the funnel calibrated as follows: Using the procedure described in Section 5, the flow rate of standard 150-mesh Turkish emery is determined. The average of five determinations (the extremes of which shall not differ by more than 0.4 s) is stamped on the bottom of the funnel. The correction factor of the unused funnel is 40.0 divided by this number. It is recommended that the factor be periodically verified by the user by determining, by the above method, the flow rate of the standard 150-mesh Turkish emery.³ If the flow rate has changed from that stamped on the instrument, the new correction factor will be 40.0 divided by this new flow rate. Before adopting the new correction factor, however, it is recommended that the cause of the change be investigated. If the flow rate has increased, it is probable that repeated use has burnished the orifice and the new correction factor may be used. A decrease in flow rate may indicate a plating of soft powder upon the orifice. This should be carefully removed with the aid of a pipe cleaner and the calibration test rerun, the new correction factor being calculated if required. It is recommended that the use of a funnel be discontinued after the flow rate of the standard sample has increased such that the time of flow is less than 37 s. The manufacturer's experience indicates that, under conditions of almost continuous daily use, a decrease in time of flow of 3 s should be expected after 5 years of service.

7. Precision and Bias

7.1 The precision of this test method is presently being determined by Subcommittee B09.02.

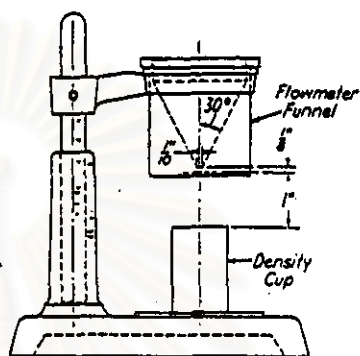
¹ This test method is under the jurisdiction of ASTM Committee B-9 on Metal Powders and Metal Powder Products and is the direct responsibility of Subcommittee B09.02 on Basic Metal Powders.

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² The powder flowmeter funnel, density cup, and stand are available from Alcan Powders and Pigments, 901 Lehigh Ave., Union, NJ 07083-7632.

³ Standardized No. 150 emery grit is no longer being sold. In those instances where the user desires to verify the correction factor and does not possess the No. 150 emery grit, the funnel may be returned to Alcan Powders and Pigments, 901 Lehigh Ave., Union, NJ 07083-7632, for re-calibration and re-certification. It is recommended that verification be done at least annually depending on frequency of use.

B 213



Metric Equivalents

in.	mm
1/16	2.54
1/8	3
1	25

FIG. 1 Flowmeter Apparatus

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Designation: B 214 - 92

Standard Test Method for Sieve Analysis of Granular Metal Powders¹

This standard is issued under the fixed designation B 214; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

1. Scope

1.1 This test method covers the determination of the dry sieve analysis of granular metal powders.

1.2 This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

- B 212 Test Method for Apparent Density of Free-Flowing Metal Powders²
- B 215 Practices for Sampling Finished Lots of Metal Powders²
- B 243 Terminology of Powder Metallurgy²
- E 11 Specification for Wire-Cloth Sieves for Testing Purposes³

3. Terminology

3.1 *Definitions*—Useful definitions of terms for metal powders and powder metallurgy are found in Terminology B 243.

4. Significance and Use

4.1 The particle size distribution of a metal powder affects its behavior in P/M processing and other applications of these materials. The test method may be part of the purchase agreement between powder manufacturer and user, or it may be an internal quality control test for either. This test method is appropriate for materials with size distributions typified by base metal powders used in powder metallurgy.

5. Apparatus

5.1 *Sieves*—A set of standard sieves selected from Table 1 of Specification E 11, or the equivalent Tyler standard sieves. The sieves shall be 8 in. (203.2 mm) in diameter and either 1 or 2 in. (25 or 50 mm) in depth and fitted with bronze, brass, stainless steel, or other suitable wire cloth. The U.S. sieves given in Table 1 of this method shall conform to Specification E 11. If Tyler standard sieves are substituted, they shall

conform to the permissible variations given in Table 1 of Specification E 11.

NOTE 1—The new U.S. Series standard sieves, adopted in 1970, are the preferred sieves to use. The old U.S. Series standard sieves and equivalent sieves manufactured by other companies, such as Tyler, may also be used if the new U.S. Series is not available. Care should be taken to make sure that sieve opening sizes are correct when performing standardization work.

5.2 *Sieve Shaker*—A mechanically operated, single eccentric sieve shaker which imparts to the set of sieves a rotary motion and tapping action of uniform speed, shall be used. The number of rotations per minute shall be between 270 and 300. The number of taps per minute shall be between 140 and 160. The sieve shaker shall be fitted with a plug to receive the impact of the tapping device. The entire apparatus shall be rigidly mounted by bolting to a solid foundation, preferably of concrete. A time switch should be provided to ensure accuracy of duration of the test.

NOTE 2—Use of a sound proof enclosure is recommended.

5.3 *Balance*—A balance having a capacity of at least 100 g and a sensitivity of 0.01 g.

6. Test Specimen

6.1 The size of the test specimen shall be 100 g for any metal powder having an apparent density greater than 1.50 g/cm³ when determined in accordance with Test Method B 212. A 50-g specimen shall be used when the apparent density of the powder is less than 1.50 g/cm³. The test specimen should be obtained in accordance with Practices B 215.

7. Procedure

7.1 Assemble the group of sieves selected in consecutive order as to size of openings, with the coarsest sieve at the top, the assembly being completed by a solid collecting pan below the bottom sieve. Place the test specimen on the top sieve and close this sieve with a solid cover. Then fasten the sieve assembly securely in a suitable mechanical sieve shaking device and operate the machine for a period of 15 min.

7.2 Remove the screened fractions from the nest of sieves by removing the coarsest sieve from the nest, gently tapping its contents to one side and pouring them upon a glazed paper. Brush any material adhering to the bottom of the sieve and frame with a soft brush into the next finer sieve. Tap the sieve just removed upside down, on the paper containing the portion that had been retained on it. Weigh this fraction to the nearest 0.1 g and remove it from the balance. Repeat this process for each sieve in the nest and

¹ This test method is under the jurisdiction of ASTM Committee B-9 on Metal Powders and Metal Powder Products, and is the direct responsibility of Subcommittee B09.02 on Base Metal Powders.

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² Annual Book of ASTM Standards, Vol 02.05

³ Annual Book of ASTM Standards, Vol 14.02.



B 214

TABLE 1 U.S. Standard Series Test Sieves and Equivalent Tyler Standard Sieves

Mesh Designation Number	Sieve Opening (μm)		
	New U.S. Series	Old U.S. Series	Tyler Series
20	850	841	833
35	—	—	417
40	425	420	—
60	250	250	295
80	180	177	175
100	150	149	147
140	106	105	—
150	—	—	104
200	75	74	74
230	63	63	—
250	—	—	53
325	45	44	44

remove the fraction collected in the pan and weigh. The sum of the weights of all the fractions shall be not less than 99 % of the weight of the test specimen. Add the difference between this sum and the weight of the test specimen (100 g or 50 g in accordance with Section 6) to the weight of the fraction collected in the pan.

NOTE 3—If the sum is less than 99 %, check the condition of the screens and pan or possible errors in weighing and repeat the test.

8. Report

8.1 Express the weights of the fractions retained on each sieve, and the weight of the fraction collected in the pan, as percentages of the weight of the test specimen to the nearest 0.1 %, and report them in the form shown in Table 2. Report any fraction that is less than 0.1 % of the weight of the test specimen as "trace." If a fraction is absent, report it as "0.0".

8.2 Interpretation of this report should be made with

TABLE 2 Form for Reporting Test Data

New U.S. Standard Series		
Particle Size (μm)	Mesh Designation No.	% By Weight
> 180	+ 80	...
≤ 180 > 150	- 80 + 100	...
≤ 150 > 106	- 100 + 140	...
≤ 106 > 75	- 140 + 200	...
≤ 75 > 45	- 200 + 325	...
≤ 45	- 325	...
Old U.S. Standard Series		
Particle Size (μm)	Mesh Designation No.	Percentage by Weight
> 177	+ 80	...
≤ 177 > 149	- 80 + 100	...
≤ 149 > 105	- 100 + 140	...
≤ 105 > 74	- 140 + 200	...
≤ 74 > 44	- 200 + 325	...
≤ 44	- 325	...
Tyler Standard Sieve Series		
Particle Size (μm)	Mesh Designation No.	Percentage By Weight
> 175	+ 80	...
≤ 175 > 147	- 80 + 100	...
≤ 147 > 104	- 100 + 150	...
≤ 104 > 74	- 150 + 200	...
≤ 74 > 44	- 200 + 325	...
≤ 44	- 325	...

reference to Specification E 11 in which the dimensional tolerances of standard sieves are specified.

9. Precision and Bias

9.1 The precision and bias that can be expected through use of this test method is currently under review by Subcommittee B09.02 on Base Metal Powders.

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APPENDICES

(Nonmandatory Information)

X1. CERTIFIED SIEVES—MASTER SET

X1.1 Sieves conforming to Specification E 11 can be obtained from the sieve manufacturers, and arrangements can be made through them to have the sieves certified by the National Institute of Standards and Technology (formerly the National Bureau of Standards).⁴ If used continually, the

sieves will, after a period of time, become less accurate and might no longer be acceptable as certified sieves. The common practice which would be considered acceptable according to this standard, would be to use the certified sieves as a master set for checking other working sets of sieves. By comparing sieve tests on the same sample, run in both the master set and the working set, a factor can be established for correcting results on the working sieves.

⁴ Contact the National Institute of Standards and Technology, Gaithersburg, MD 20899.

X2. MATCHED SIEVES

X2.1 The use of a matched set of sieves, established through use of a standard powder, is recommended when

closer correlation of tests between supplier and consumer is desired.

X3. SIEVE SERIES GUIDELINES

X3.1 Suggested combinations of sieves are given in Table X3.1 for several nominal mesh size metal powders.

TABLE X3.1 Suggested Sieve Series for Metal Powders

Nominal Powder Mesh Size	20	40	80	100	140	200	325
New U.S.	20	✓	✓				
	40	✓	✓	✓			
	80	✓	✓	✓			
Sieve	80			✓			
	100	✓	✓	✓	✓		
Series	140		✓	✓	✓	✓	
	200	✓	✓	✓	✓	✓	✓
(Mesh Sizes)	230						✓
	325		✓	✓	✓	✓	✓
	Pan	✓	✓	✓	✓	✓	✓

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Designation: B 527 - 93

Standard Test Method for Determination of Tap Density of Metallic Powders and Compounds¹

This standard is issued under the fixed designation B 527; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript symbol (e) indicates an editorial change since the last revision or reappraisal.

1. Scope

1.1. This test method specifies a method for the determination of tap density (packed density) of metallic powders and compounds, that is, the density of a powder that has been tapped, to settle contents, in a container under specified conditions.

1.2 This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

- 2.1 *ASTM Standards*:
 B 212 Test Method for Apparent Density of Free-Flowing Metal Powders²
 B 215 Practice for Sampling Finished Lots of Metal Powders²
 B 243 Terminology of Powder Metallurgy²
 B 329 Test Method for Apparent Density of Powders of Refractory Metals and Compounds by Scott Volumeter²
 B 417 Test Method for Apparent Density of Non-Free-Flowing Metal Powders²
 B 703 Test Method for Apparent Density of Metal Powders Using the Arnold Meter²

3. Significance and Use

3.1 This test method covers the evaluation of the tapped density physical characteristic of metallic powders and compounds. The degree of correlation between the results of this test method and the quality of powders in use will vary with each particular application and has not been fully determined.

4. Apparatus

4.1 *Balance*, of appropriate capacity and accuracy to satisfy the requirements shown in Table 1.

4.2 *Graduated Glass Cylinder*³, calibrated to contain 100 cm³ at 20°C, the height of the graduated portion being approximately 175 mm. The graduations shall be at 1 cm³

¹ This specification is under the jurisdiction of ASTM Committee B-9 on Metal Powders and Metal Powder Products and is the direct responsibility of Subcommittee B09.03 on Refractory Metal Powders.

Current edition approved Dec. 15, 1993. Published April 1994. Originally published as B 527 - 70. Last previous edition B 527 - 85 (1991).⁴

² *Annual Book of ASTM Standards*, Vol 02.05.

³ Coming No. 3046, Pyrex brand has been found suitable.

TABLE 1 Accuracy and Capacity of Balance

Cylinder Capacity, cm ³	Apparent Density, g/cm ³	Mass of Test Portion, g
100	>1	100 ± 0.5
100	<1	50 ± 0.2
25	>7	100 ± 0.5
25	2 to 7	50 ± 0.2
25	<2	20 ± 0.1

intervals, thus allowing a measuring accuracy of ± 0.5 cm³. For apparent densities over 4.0 g/cm³, do not use the 100 cm³ cylinder.

4.2.1 Alternatively, the following may be used:

4.2.1.1 *Graduated Glass Cylinder*, calibrated to contain 25 cm³ at 20°C, the height of the graduated portion being approximately 135 mm. The graduations shall be at 0.2 cm³ intervals.

4.2.1.2 A 25-cm³ cylinder shall be used for powders of apparent density higher than 4 g/cm³, in particular for refractory metal powders, but may also be used for powder of lower apparent density.

4.3 *Tapping Apparatus*,⁴ which permits the tapping of the graduated cylinder against a firm base. The tapping shall be such that a densification of the powder can take place without any loosening of its surface layers. The stroke shall be 3 mm (0.118 in.) and the tapping frequency shall be between 100 and 300 taps/min. An example of a tapping apparatus is shown in Fig. 1.

5. Test Specimen

5.1 For the quantities of powder required for each test, see Table 1. Obtain test powder samples according to Practices B 215.

5.2 In general, the powder should be tested in the as-received condition. In certain instances the powder may be dried. However, if the powder is susceptible to oxidation, the drying shall take place in a vacuum or in inert gas. If the powder contains volatile substances, it shall not be dried.

5.3 The test shall be carried out on three test samples.

6. Procedure

6.1 Clean the inside the wall of the graduated cylinder (5.2) with a suitable clean brush or, if necessary, by rinsing with a solvent, such as acetone. If a solvent is used, thoroughly dry the cylinder before reuse.

⁴ The following have been found suitable: Shandon Southern Instruments, Inc., Tap-Pak Volumeter Model JEL-ST2 (Manufactured by J. Engelmann A.G. of Ludwigshafen 9. Rh. West Germany), 515 Broad Street, Sewickley, PA 15143; Vankel Industries, Vanderkamp Tap Density Taper, 36 Meridian Road, Edison, NJ 08820; Quantachrome Corp., Dual Autotap, 6 Aerial Way, Syosset, NY 11791.

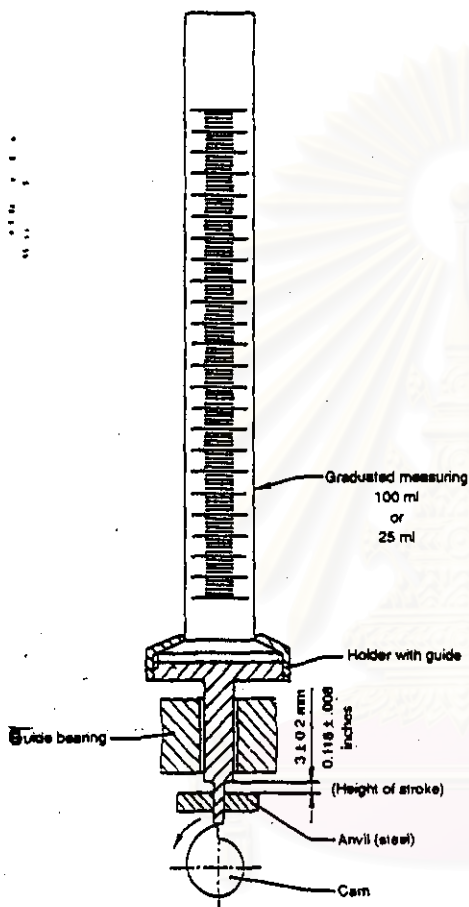
 B 527


FIG. 1 Example of Tapping Apparatus

6.2 Weigh, to the nearest 0.1 g, the mass of the test portion as indicated in Table 1, using a balance (4.1).

6.3 Pour the test portion into the graduated cylinder. Take care that a level surface of the powder is formed. Place the cylinder in the tapping apparatus (4.3). Tap the cylinder until no further decrease in the volume of the powder takes

place (see Note 1).

NOTE 1—In practice, the minimum number of taps, N , such that no further change in volume takes place would be determined. For all further tests on the same type of powder, the cylinder would be subjected to $2N$ taps, except where general experience and acceptance have established a specific number of taps (no less than N taps) as being satisfactory. For fine refractory metal powders, 3000 taps has been found to be satisfactory for all sizes.

6.4 If the tapped surface is level, read the volume directly. If the tapped surface is not level, determine the tap volume by calculating the mean value between the highest and the lowest reading of the tapped surface. Read the final volume to the nearest 0.5 cm³ when using a 100 cm³ cylinder and to the nearest 0.2 cm³ when using a 25 cm³ cylinder.

7. Calculation

7.1 The tap density is given in the following equation

$$P_t = \frac{M}{V}$$

where:

P_t = tap density, g/cm³,
 M = mass of powder, g, and
 V = volume of tapped powder, cm³.

8. Report

8.1 Report the following information:

- 8.1.1 Reference to this test method,
- 8.1.2 All details necessary for identification of the test sample,
- 8.1.3 The drying procedure, if the powder has been dried,
- 8.1.4 Cylinder capacity; mass of test portion and method used,
- 8.1.5 The result obtained,
- 8.1.6 All operations not specified in this test method or regarded as optional,
- 8.1.7 Details of any occurrence that may have affected the result.

9. Precision and Bias

9.1 Precision and bias cannot be stated at this time because this test method covers a broad range of powders and associated densities.

10. Keywords

10.1 apparent density; bulk density; density; density ratio; metal powders; packed density; powder metallurgy; tap density

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Designation: C 373 - 88 (Reapproved 1994)

Standard Test Method for Water Absorption, Bulk Density, Apparent Porosity, and Apparent Specific Gravity of Fired Whiteware Products¹

This standard is issued under the fixed designation C 373; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers procedures for determining water absorption, bulk density, apparent porosity, and apparent specific gravity of fired unglazed whiteware products.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Significance and Use

2.1 Measurement of density, porosity, and specific gravity is a tool for determining the degree of maturation of a ceramic body, or for determining structural properties that may be required for a given application.

3. Apparatus and Materials

3.1 *Balance*, of adequate capacity, suitable to weigh accurately to 0.01 g.

3.2 *Oven*, capable of maintaining a temperature of $150 \pm 5^\circ\text{C}$ ($302 \pm 9^\circ\text{F}$).

3.3 *Wire Loop, Halter, or Basket*, capable of supporting specimens under water for making suspended mass measurements.

3.4 *Container*—A glass beaker or similar container of such size and shape that the sample, when suspended from the balance by the wire loop, specified in 3.3, is completely immersed in water with the sample and the wire loop being completely free of contact with any part of the container.

3.5 *Pan*, in which the specimens may be boiled.

3.6 *Distilled Water*.

4. Test Specimens

4.1 At least five representative test specimens shall be selected. The specimens shall be unglazed and shall have as much of the surface freshly fractured as is practical. Sharp edges or corners shall be removed. The specimens shall contain no cracks. The individual test specimens shall weigh at least 50 g.

5. Procedure

5.1 Dry the test specimens to constant mass (Note) by

heating in an oven at 150°C (302°F), followed by cooling in a desiccator. Determine the dry mass, D , to the nearest 0.01 g.

NOTE—The drying of the specimens to constant mass and the determination of their masses may be done either before or after the specimens have been impregnated with water. Usually the dry mass is determined before impregnation. However, if the specimens are friable or evidence indicates that particles have broken loose during the impregnation, the specimens shall be dried and weighed after the suspended mass and the saturated mass have been determined, in accordance with 5.3 and 5.4. In this case, the second dry mass shall be used in all appropriate calculations.

5.2 Place the specimens in a pan of distilled water and boil for 5 h, taking care that the specimens are covered with water at all times. Use setter pins or some similar device to separate the specimens from the bottom and sides of the pan and from each other. After the 5-h boil, allow the specimens to soak for an additional 24 h.

5.3 After impregnation of the test specimens, determine to the nearest 0.01 g the mass, S , of each specimen while suspended in water. Perform the weighing by placing the specimen in a wire loop, halter, or basket that is suspended from one arm of the balance. Before actually weighing, counterbalance the scale with the loop, halter, or basket in place and immerse in water to the same depth as is used when the specimens are in place. If it is desired to determine only the percentage of water absorption, omit the suspended mass operation.

5.4 After the determination of the suspended mass or after impregnation, if the suspended mass is not determined, blot each specimen lightly with a moistened, lint-free linen or cotton cloth to remove all excess water from the surface, and determine the saturated mass, M , to the nearest 0.01 g. Perform the blotting operation by rolling the specimen lightly on the wet cloth, which shall previously have been saturated with water and then pressed only enough to remove such water as will drip from the cloth. Excessive blotting will introduce error by withdrawing water from the pores of the specimen. Make the weighing immediately after blotting, the whole operation being completed as quickly as possible to minimize errors due to evaporation of water from the specimen.

6. Calculation

6.1 In the following calculations, the assumption is made that 1 cm³ of water weighs 1 g. This is true within about 3 parts in 1000 for water at room temperature.

6.1.1 Calculate the exterior volume, V , in cubic centimetres, as follows:

$$V = M - S$$

¹ This test method is under the jurisdiction of ASTM Committee C-21 on Ceramic Whitewares and Related Products and is the direct responsibility of Subcommittee C21.03 on Fundamental Properties.

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6.1.2 Calculate the volumes of open pores V_{OP} and impervious portions V_{IP} in cubic centimetres as follows:

$$V_{OP} = M - D$$

$$V_{IP} = D - S$$

6.1.3 The apparent porosity, P , expresses, as a percent, the relationship of the volume of the open pores of the specimen to its exterior volume. Calculate the apparent porosity as follows:

$$P = [(M - D)/V] \times 100$$

6.1.4 The water absorption, A , expresses as a percent, the relationship of the mass of water absorbed to the mass of the dry specimen. Calculate the water absorption as follows:

$$A = [(M - D)/D] \times 100$$

6.1.5 Calculate the apparent specific gravity, T , of that portion of the test specimen that is impervious to water, as follows:

$$T = D/(D - S)$$

6.1.6 The bulk density, B , in grams per cubic centimetre, of a specimen is the quotient of its dry mass divided by the exterior volume, including pores. Calculate the bulk density as follows:

$$B = D/V$$

7. Report

7.1 For each property, report the average of the values obtained with at least five specimens, and also the individual values. Where there are pronounced differences among the individual values, test another lot of five specimens and, in addition to individual values, report the average of all ten determinations.

8. Precision and Bias

8.1 This test method is accurate to $\pm 0.2\%$ water absorption in interlaboratory testing when the average value recorded by all laboratories is assumed to be the true water absorption. The precision is approximately $\pm 0.1\%$ water absorption on measurements made by a single experienced operator.

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

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