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APPENDICES

APPENDIX A

Sample Preparation Procedure

I. Chemical Analysis by X-ray Fluorescence

Apparatus :

1. Grinder, Machine macina smalto, Rapid Mill
2. Fusion bead casting machine, Claisse Fluxer BIS ! 10
3. Ceramic crucible
4. Siemen X-rays Fluorescence Spectrometer

Procedure :

1. Grind sample to fine powder (-125 µm) about 10 gm.
2. Dry sample in oven at 110 ± 5 °C for 22 hrs.
3. Accurately weigh 2 gm. Of dried sample in ceramic crucible and transfer into furnace for loss of ignition test.
4. Fused sample was prepared by accurately weigh 1.000 gm. Of sample and accurately weigh 5 gm. Of flux (100B or Lithium Tetraborate), then mixed them together in platinum crucible and fix crucible with Fusion Machine.
5. Run fusion machine with suitable program for each sample type which can get the good cast bead.
6. Take cast bead into the sample holder in X-ray Fluorescence spectrometer (XRF).
7. Run XRF for chemical analysis.
8. The result was reported in element oxide.

II. Mineralogical Analysis by XRD

Prior to whole rock XRD analysis, a portion < 125 μm. powder was micronised under unoriented mounts. The micronised powder was back-loaded into a standard alumina sample holder and analysed using a Philip X-ray diffractometer model no. PX 1023, type PW 1730/10, Cu-K radiation at an operating voltage of 40 kVand current of 30 mA. Sample mounts were examined over an angular range of 2-60° and speed of 2°/ minute.

The < 2 μm. fraction was analysed as a back-loaded powder mount under identical condition to the whole rock powder. In addition (to aid the identification of clay mineral species), an oriented mount of the < 2 μm. fraction was prepared by sedimentation of material onto a glass disc. Air-dried and glycol-solvated oriented mounts were examined over angular range of 2-60 ° 2θ at a scanning speed of 2° / minute.

III. Microstructure Analysis by SEM and TEM

Samples Preparation Technique

1. Disperse ball clay (-60# mesh) in Calgon (Sodium hexametaphosphate) 0.05 % (weight by volume) by using Lightnin mixer for 20 min.
2. Drop the suspension in 1 - 3 drops in absolute ethanol ¾ of test tube.
3. Disperse it with ultrasonic probe.
4. For investigating by TEM use 1 drop of the solution in topic 3. on the grid. Dry it in the desiccator.
5. For scanning by SEM drop 2 drops of the solution in topic 3. On the stud. After drying it in the desiccator, sputtering the sample on the stud with gold for 180 seconds (3 min.)

IV. Wet Sieve for Residue Analysis

Apparatus :

1. Sieve No. 120 #, 200 # and 325 # mesh, Endecotts, Specification A.S.T.M.
2. Variable speed stirrer, Lightnin< G 3U05R
3. Container

Procedure :

1. Approximately weigh 300 gm. Of sample (dry weight) to container.
2. Add 1 liter of water and allow to slake.
3. Agitate the slurry with high speed stirrer until completely dispersion of sample.
4. Transfer dispersed sample, without loss, to sieve series with coarse sieve is on top of finer sieve.
5. Transfer residue on each sieve to each container with water.
6. Waiting for residue in container to settle, rinse water out.
7. Place all container in oven for drying at 110 °C until thoroughly dry approximately 3 hours.
8. Weigh the residue on sieve 120 #, record as A.
9. Include residue on sieve 200 # with residue in topic 8, record as B.
10. Include residue on sieve 325 # with residue in topic 9, record as C.
11. Calculate % Cumulative residue on sieve as follow :

% Residue sieve 120 # = (A x 100) / Weight of sample
% Residue sieve 200 # = (B x 100) / Weight of sample
% Residue sieve 325 # = (C x 100) / Weight of sample

Where :

A = Weight of dried residue on 120 #

B = Weight of dried residue on 120 # + 200 #

A = Weight of dried residue on 120 # + 200 # + 325 #

V. Particle Size Distribution Analysis

Apparatus :

1. Sedigraph 5100, Micromeritics
2. Variable Speed Mixer, Heidolph RZR 2041
3. Ultrasonic Bath, Astrason

Procedure :

1. Calculated dry weight 8 gm. Of sample from slip.
2. Weigh slip as calculated result.
3. Add 0.05 % sodium hexametaphosphate as calculated result, so

   100 – (weight of slip – 8)
4. Agitate the slurry by mixer about 15 min.
5. Screen slurry through 200 # sieve.
6. Transfer 50 ml. Of filtrate to beaker and put it on ultrasonic bath for about 10 min.
7. 7Pour that filtrate in mixing chamber of Sedigraph.
8. Run Sedigraph for analyze Particle Size Distribution.

VI. Slip Preparation for Viscosity and Casting Properties Test

Apparatus :

1. Torsion Viscometer, Gallenkamp. Or Brookfield Viscometer model RV DVII^{+}
2. Half-moon plaster mold, Baroid filter
3. Variable Speed Mixer, Lightnin, G 3U05R

Procedure :

1. Approximate weigh 100 gm. Of sample (dry weight) in a container.
2. Add 1 liter of DI. Water and allow to slake.
3. Adjust slip to give specific gravity at each requirement for each ball clay samples.
4. Add 0.5 gm/ml sodium silicate slowly and obtain the flow enough to sieve through 120 # mesh.
5. Transfer it to weight in the container record the weight to calculate amount of used sodium silicate.
6. Stir it with the mixer and gradually added sodium silicate till the Overswing is 355-360 and lowest Thixo at 1 min. for fully deflocculation test.
7. For deflocculation response test when sieve through 120 # mesh. Start to test as in the test method 5.5.1.1.
8. In case of humic acid added ball slip, humic acid is first dispersed in DI water for 30 min, then pour the ball clay sample in the container and allow to slake, after that follow by the next step in topic 3.

APPENDIX B

Test Method for Soluble salts Analysis

Soluble salts analysisuse Standard Methods for Water and Wastewater 18th Edition 1992; Edited by Arnold E. Greenberg, Lenor S. Clesceri, Andrew D. Eaton.

Specific conductance: Measure with CDM Conductivity Meter by using Ionalyzer (Orion Research model 701 A/ digital)

Ca: To measure amount of Ca use complexometric method by tritration with disodium dihydrogen ethylenediamene tetraacetate (Na_2EDTA) 0.020 N. and use Murexide as indicator in base solution while hving hydroxylamine prevent interference effect from the oyher metals.

Hardness: Use complexometric method by titrating with Na_2EDTA in base solution (adding NH_4OH) and use hydroxylamine for preventing interference effects and has Eriochrome Black T as indicator.

Cl: Use Argentometric method by presenting Potassium chromate (K_2CrO_4) as indicator and titrate it with conc. $AgNO_3$ 1,000 mg/liter

HCO_3: Use tritration method by tritrating with H_2SO_4 and there is methyl orange as indicator.

Fe: Use Atomic Absorption Spectrometric Method.

Mn: Use Atomic Absorption Spectrometric Method.

SO_4: Measure by Auto analyzer II using Automated Methylthymol Blue Method.

NO_3, NO_2: Measure by Auto analyzer II using Automated Cadmium Reduction Method.

F: Measure by Ionalyzer (Orien Research model701A/digital) using Ion Selective Method.

Total dissolved solids: By calculating conductive (multiply by 0.65 ; researched value)

Mg: Using (meq. Of Total hardness – meq.of Ca) x 50.

Na: Using Flame Emission Photometric Method. Measure by Flame emission photometer (Klina Flame),

K: Using Flame Emission Photometric Method. Measure by Flame emission photometer (Lisabio PHF 90D)

CO_3: Using tritration method by H_2SO_4 ($\approx 0.020\ N$) and phenolphthalene as indicator.

Non-carbonate hardness : Calculating by (meq. Of Total hardness – meq. Of Ca) x 50

Biography

Varangkana Klinsukol was born on March 21, 1963 in Bangkok. She received a bachelor degree in materials science from Faculty of Science, Chulalongkorn University in 1985 and bachelor degree in Business Administration (general management) from Sukhothai Thammathirat Open University in 1995. She enrolled for her master study in March 1996 and completed the program in October 1998.