### **CHAPTER 4**

#### METHODS OF ATTACK

This study composes of 11 ball clay samples from local and imported sources then classify them into three groups by using performance in casting process. It consisted of the characterization and identification of the fundamental features of ball clays as; chemical analysis by X-ray Fluorescence (XRF), mineral analysis by X-ray Diffraction (XRD), microstructure by both Scanning Electron Microscope (SEM) and Transmitted Electron Microscope (TEM), particle size distribution analysis by x-ray sedimentomerter (Sedigraph). The especially method to investigate in their organic matter and soluble salts is by applying soil science technique to elucidate the organic matter and water and waste water analysis to determine soluble salts that is different to conventional ceramic study method. The eminent method that uses in this work is to identify the humic substance that extracted from the samples by NMR spectroscopy.

After characterize these ball clay samples, they were prepared to ball slips for rheology and casting properties study. The rheology properties in this work concentrated on deflocculation response, gelation and relative gel-strength, shear response and had some temperatures effect for the basic information by using Brookfield viscometer RV DVII<sup>+</sup>. Casting properties were done by 2 methods; plaster mold and Baroid filter press

The next step is to study the effect of organic matter by adding extractable humic acids and commercial to MT ball clay then preparing to ball slip and study the rheology and casting properties.

#### Method of Attack Chart

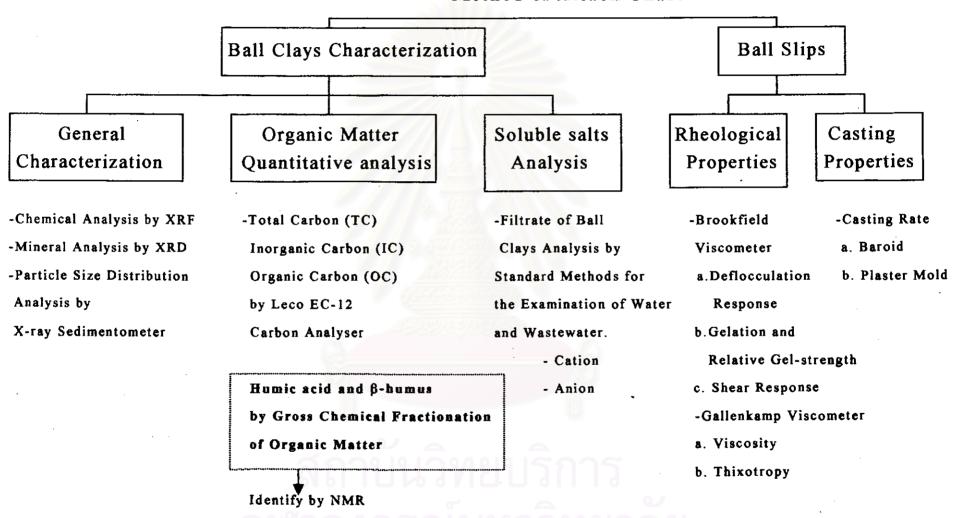


Fig. 4.1 Method of attack chart

### 4.1 Ball Clay Selection.

We can separate ball clay that used in Thai sanitaryware industry into local and imported clay. So the ball clays have been chosen to study in this research can be separated into three groups by using the importance characteristic of sanitaryware body, and casting properties. The three groups of ball clay are:

- Group I Local ball clays that have good rheological and casting properties.
  - MAE SAN (MS)
  - MAE VIT WHITE (MVW)
  - MAE TILE (MT).
- Group II Imported ball clays that have good rheological and casting properties.
  - from United Kingdom
    - SB-75 product of WBB.
    - HYCAST VC (HVC) product of ECC.
  - -from United State of America
    - REX product of United Clay.
    - BANDY BLACK (BB) product\_of (H.C. SPINKS)
- Group III Local ball clays that have inferior rheological and casting properties.
  - JAE KORN (JK) from tumbol Thung Phung, amphur Jae Hom, Lampang province.
  - Kok Kharm (KK) from tumbol Kok Kharm, amphur Ron Phibun, Nakhon Si thammarat province.
  - Wang Nhue (WN) from amphur Wang Nua, Lampang province.
  - Parn (PC) from amphur Parn, Chiang Rai province.

JK is now being used in small percentage in sanitaryware because of its cost. But KK, WN and PC are used for tile industry<sup>(3,4)</sup>.

## 4.2 Characterizing Features of Ball Clay

General characterization of ball clay are consists of

- chemical analyses reported in the traditional weight percent of oxide format: SiO<sub>2</sub>, TiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, CaO, MgO, K<sub>2</sub>O, Na<sub>2</sub>O, P<sub>2</sub>O<sub>5</sub>, MnO, LOI. by using X-ray Fluoresence (XRF).
- Mineral Phases Identification: A thorough qualitative and quantitative identification by means of X-ray Diffraction.
- Microstructure investigation by Scanning Electron Microscope (SEM) and Transmitted Electron Microscope (TEM).
- Particle size distribution: Screen residue on 120 #, 200 # , 325 # mesh and particle size distribution analysis were obtained from standard laboratory sieves and Micromeritics Sedigraph model 5100 D particle size analyzer at the range of  $-20~\mu m 0.2~\mu m$ .

Colloid Modifiers analysis method

- Organic matter in ceramic method is practically in the form of LOI. and Total carbon, organic, and inorganic carbon.
  - Soluble salts are mentioned in the form of soluble sulfate.

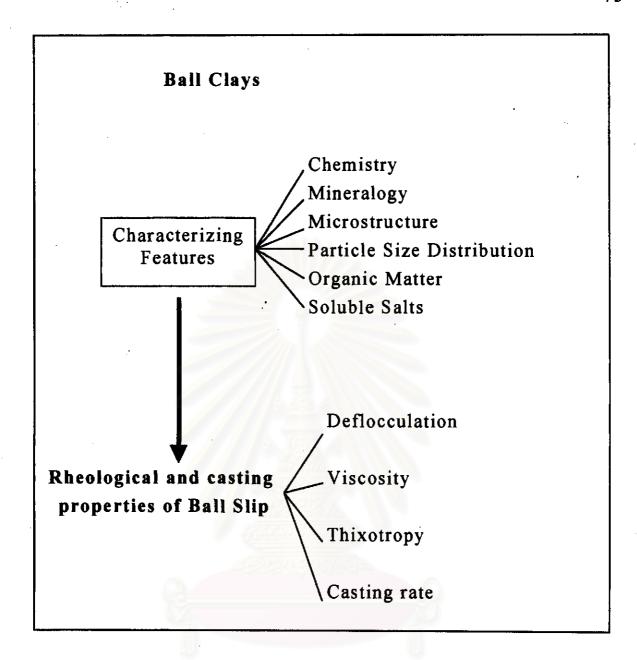


Fig. 4.2 Ball Clays Characterization Chart

# 4.3 Quantitative Determination of Organic Matter

Determination of ball Clay Organic matter (28):

- Evaluate organic matter in quantity of Total Carbon (TC), Organic carbon (OC) by combustion in oxygen atmosphere at 1000°C by Leco EC-12 Carbon Analyser and deduct TC by OC and resulting the Inorganic Carbon (IC).

- Quantitative analysis of Humic substance<sup>(29)</sup> as Humic acid and  $\beta$ -humus (a part of Fulvic acid) by Gross Chemical Fractionation of Organic Matter.

-Identify Humic substance by Nuclear Magnetic Resonance (NMR) using CP/MAS<sup>13</sup>C NMR. (30,31)

# 4.4 Soluble Salts Analysis (38)

Using filtrate of Ball clay sample in deionized water to have soluble salts analysis by Soluble Salt Analytical Procedure follow by Standard Methods for the Examination of Water and Wastewater. Resulting this elements:

Ca, Mg, Na, K, Fe, Mn, Cl, SO<sub>4</sub>, HCO<sub>3</sub>, NO<sub>2</sub>, NO<sub>3</sub>, F, Total dissolved solids, Total hardness as CaCO<sub>3</sub>, Noncarbonata hardness in the unit of milligram per kilogram.

### 4.5 Rheological and Casting Properties Evaluation

Rheological and casting properties are reported in the form of deflocculation response, flow behavior by Brookfield and Gallenkamp viscometer, casting rate by baroid and plaster mold.

-Deflocculation behavior with selected specific gravity (1.55 gm/ml for local ball clays and 1.63 for imported ball clays). Measuring the viscosity at each content of deflocculant (Na<sub>2</sub>SiO<sub>3</sub>) by Brookfield viscometer in centipoise till fully deflocculation occurs.

-Flow behavior study

Brookfield viscometer resulting

a) Gelation curve for 15 min. and Relative gel-strength that obtain by leaving ball slip to gel in 5 min. then start to collect the viscosity again.

b.) Shear response in the terms of Shear rate - Shear stress curve and shear rate - viscosity curve measured by small sample adapter of Brookfield viscometer

Gallenkamp viscometer report Thixotropy in Overswing and Thixo 1 min.

-Casting rate measure by Baroid and Plaster mold methods. For Baroid filtration casting rate use 58 Psi. 30 min. and report wet cake weight, dry cake weight, % humidity and weight of filtrate. And Plaster mold casting rate resulting in the thickness of mm. per 30 min.

And the last part of this work to proof the effect of humic acid on the rheology and casting properties by adding the selected extractable humic acid(HVC and BB Humic acid) in MT ball slip compare to the addition of commercial humic acid. In view of the reasons to improve ball clay rheology and casting properties.