CHAPTER 3

Materials and Experimental procedure

3.1 Raw materials

3.1.1 Perlite

Perlite is a generic term for naturally occurring siliceous rock which sets apart from other volcanic glasses is that when heated to a suitable point in its softening range, its expands from four to twenty times its original volume.

The expansion is due to presence of two to six percents combined water in the crude perlite rock. When quickly heated to above 871°C, the crude rock pops similar to popcorn as a combined water vaporizes and creates countless tiny bubbles which account for the amazing light weight and other exceptional physical properties of expanded perlite. The expanded perlite is snowy white to grayish color. While the crude perlite may range from transparent light gray to glossy black.

Since perlite is a form of natural glass, it is classified as chemically inert and has a pH of approximately 7.

Chemical composition of perlite contains SiO_2 72.7% (70.3-75.1%), Al_2O_3 12.9% (12.2-13.7%), Fe_2O_3 3.4%, TiO_2 0.9%, MgO 0.6%, CaO 0.6%, K_2O 0.2% and LOI 3.0%. Physical properties of perlite are shown in Table 3.1.

There are many uses for perlite. These uses can be broken down into three general categories: construction application, horticultural application, and industrial application.

Additional applications include its use as an abrasive in soaps, cleaners, and polishes *(22)*.

Table 3.1 Typical physical characteristics of perlite *(22)*

Color	white
Refractive Index	1.5
Free Moisture, Maximum	0.50%
pH (of water slurry)	6.5 - 8.0
Specific Gravity	2.2 - 2.4
Bulk Density (loose weight)	As desired but usually in the 2-25 lb/ft^3 range (32-400 kg/m^3)
Mesh Size Available	As desired, 4-8 mesh and finer
Softening Point	1600-2000°F (871-1093°C)
Fusion Point	2300-2450°F (1260-1343°C)
Solubility	- Soluble in hot concentrated alkali and HF - Moderately soluble (<10%) in 1N NaOH - Slightly soluble (<3%) in mineral acids (1N) - Very slightly soluble (<1%) in water or weak acids

3.1.2 Diatomite

Diatomite is a siliceous sedimentary material formed by the accumulation of the skeletal remains of microscopic plants commonly known as diatoms *(23)*. Diatoms are microscopic members of the algae family consisting of many thousands of species and are between 1-1000 microns, typically 50-100 microns in size. Their skeletons are composed of silica and they occur in a wide variety of shape. These include cylindrical, rod-like and star shape form, typically with a perforated surface and hollow interior.

The diatoms are composed of an amorphous form of silica containing a small amount of microcrystalline material. Diatomite is chemically stable and inert. Because of the open structure of the diatom skeletons. Diatomite is a lightweight rock (SG 1.95) and quite hard (Hardness 4.5-5.0) *(24)*.

The unique intricate structure of diatomite particles and its chemical stability make it desirable as a filter aid. Currently over half of the world diatomite production is used as a filter aid in beverage industries to help clarify wine, beer and fruit juices and in water purification systems. The structure of the diatom shell results in a low bulk density powder, with 90% of its volume composed of interconnected voids and pores. Dry bulk density ranges from 128-320 kg/m^3 with absorbency capabilities 2-2.5 times its dry weight, yet it does not readily absorb water from air. The high pore and void volume provide excellent permeability and filtration capacity, capable of removing particles less than 0.5 μm from liquids *(23)*.

Commercial diatomite contains 85-94% SiO_2 plus 1-7% Al_2O_3, 0.4-2.5% Fe_2O_3, 0.1-0.5% TiO_2, 0.03-0.2% P_2O_5, 0.3-3% CaO, 0.3-0.9% K_2O plus organic matter, soluble salts (0.1-0.2%) and various rock-forming minerals. For physical characteristics of diatomite are shown in Table 3.2 *(24)*.

Table 3.2 Typical physical characteristics of diatomite *(23)*

Wet Weight (kg/m^3)	256-336
Loose Weight (kg/m^3)	128-320
pH	7.0
Surface Area (M^2/g)	10-20
Color	grey, buff, white, pink
Refractive Index	1.45-1.50
Specific Gravity	2.1-2.3
Oil Absorption (lbs/100 lbs)	100-215
Porosity (%)	65-85
Water Absorption (% by wt)	86-240
Median Pore size (μm)	1.5-22.0
% Soluble	0.10-0.15
Hardness	4.5-5

For filtration uses, natural grade diatomite is calcined by heat treatment in gas or fuel oil fired rotary calciners, with or without a fluxing agent. Typical calciner operating temperatures range from 650-1200°C.

The method for treatment diatomite waste from beverage industry is described in Fig 3.1.

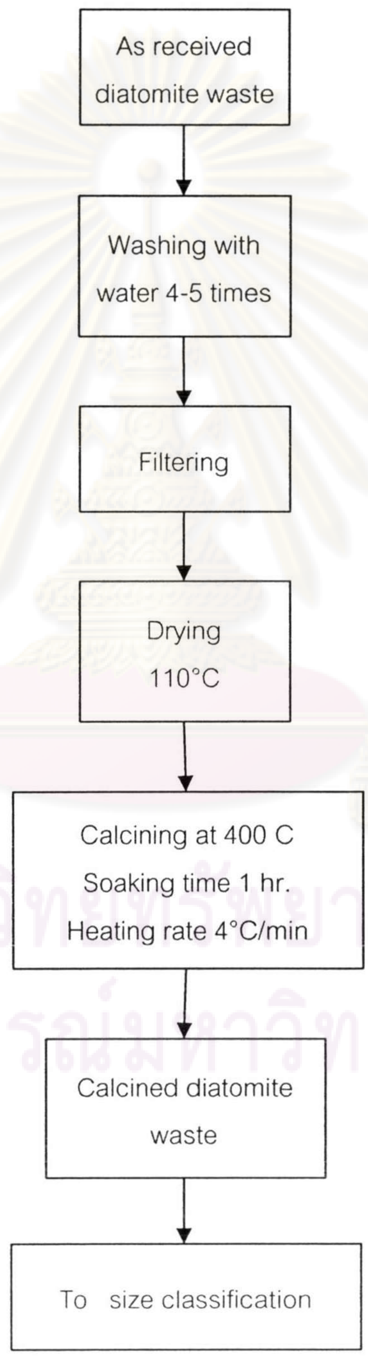

Fig 3.1 Flow chart for treatment of diatomite waste

3.1.3 Silica

The silica employed was synthesized from rice husk which was a high purity silica whose composition is 98.10% SiO_2, 0.91 Al_2O_3, 0.04% Na_2O, 0.1-0.5% K_2O, 0.02% MnO_2, 0.83% CaO, 0.03% MgO. The process flow chart for silica synthesis from rice husk is shown in Fig. 3.2 *(25)*.

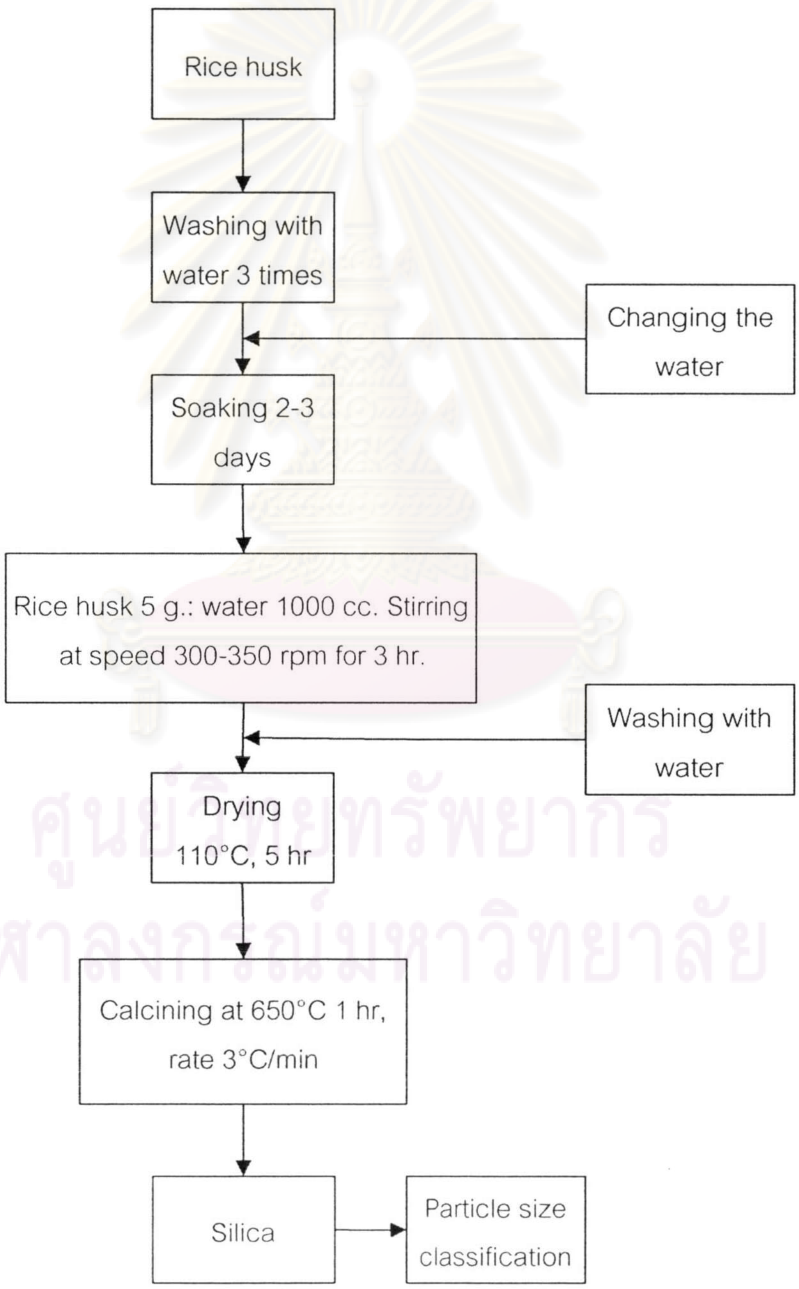

Fig 3.2 Process for synthesis of silica from rice husk *(25)*

3.1.4 Pumice

Pumice is a light, porous type of pyroclastic igneous rock. It is formed during explosive volcanic eruptions when liquid lava is ejected into the air as a froth containing masses of gas bubbles. As the lava solidifies, the bubbles are frozen into the rock. It is considered a glass because it has no crystal structure. Pumice varies in density according to the thickness of the solid material between the bubbles; many samples float in water. It is widely used to make lightweight concrete and as an abrasive, especially in polishes and cosmetic exfoliants *(26)*.

Typical chemical property of pumice contains SiO_2 70.5%, Al_2O_3 13.5%.

Table 3.3 Typical physical characteristics of pumice *(26)*

Bulk Density (g/cm3)	very low
Mohs Hardness @20°C	5.5
Specific Gravity (g/cc)	2.35
pH	7.2
Particle Shape	Irregular
Loss On Ignition	5%

3.2 Characterization of raw materials

3.2.1 Particle size distribution and shape of raw materials

The mean particle size, the size distribution and shape were investigated under a scanning electron microscope (SEM) (JEOL: JSM 6400). The average particle size and shape was determined by ASTM: F 1877-98.

3.2.2 Mineral composition

The crystalline phase compositions were identified by X-ray diffractometer (XRD) (Bruker, D8-Advance). The samples were examined with angular ranges from 10 to 70 degree 2-theta and a step scan of 3 degree per minute.

3.2.3 Chemical analysis

The chemical compositions of the raw materials were identified by X-ray fluorescence (ARL 9400).

3.3 The procedure of preparation of polishing agents

Particle size classification: The raw powders were graded according to the range of particle size and the following ranges were selected. The polishing agents were prepared following Fig 3.3.

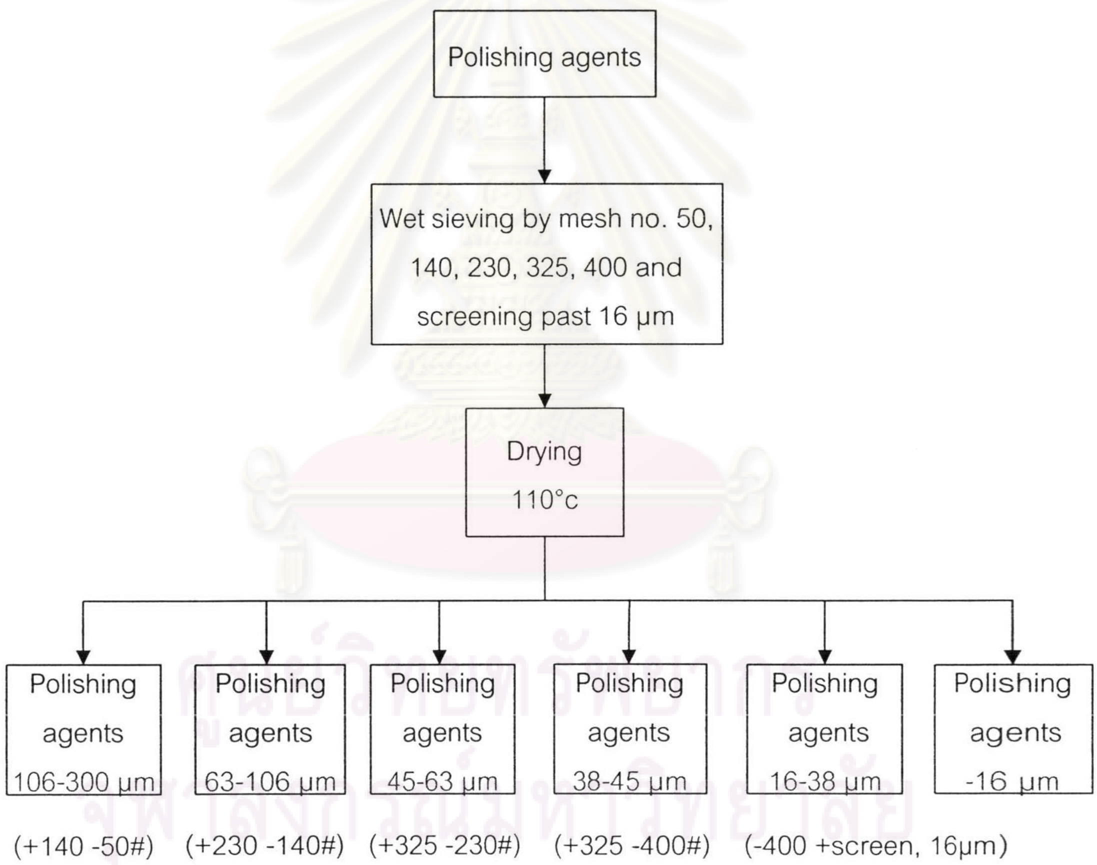

Fig 3.3 Flow chart for the preparation of polishing agents

The average particles sizes of polishing agents for the preparation of prophylaxis paste are shown in Table 3.4

Table 3.4 Average particle size of polishing agents.

Polishing agent	Average particle size (μm)
Nupro	22.22
Diatomite 505, 106-300 μm	103.44
Diatomite 505, 45-63 μm	40.42
Diatomite 505, -16 μm	10.48
Diatomite hyflow, 45-63 μm	39.10
Diatomite hyflow, -16 μm	13.51
Calcined diatomite waste, 106-300 μm	121.21
Calcined diatomite waste, -16 μm	12.66
Perlite, 106-300 μm	97.41
Perlite, -16 μm	12.90
Silica, 106-300 μm	75.16
Pumice, -16 μm	16.26

3.4 The preparation of prophylaxis paste

The preparation of prophylaxis paste was performed by prepared polishing/cleaning agents with some solvents into a homogeneous paste of proper consistency. All the ingredients used must be safe and hygienic with regard to A-A-54426. As the reference, Nupro prophylaxis paste was chosen for property comparison.

3.4.1 Prepared polishing agents

(a) Perlite AP-120 (Expanded perlite)
(Thai Perlite Technology CO., Ltd.)

(b) Diatomite 505 (Celite Corporation, Food chemicals grade)

(c) Diatomite hyflow (Celite Corporation, Food chemicals grade)

(d) Calcined diatomite waste (Pathum Thani Brewery CO., Ltd.)

(e) Silica

(f) Pumice

(g) Nupro prophylaxis paste (Control) (Dentsply Professional)

3.4.2 <u>Solvents and surfactants</u>

(a) Polyethylene glycol 400 (PEG 400) (EAC, The East Asiatic (Thailand) Public Company Limited)

Formula: $H\text{-}[OCH_2CH_2\text{-}\}_nOH$

Solubility: Polyethylene glycol 400 can be dissolved in water to form clear solutions and are soluble in many organic solvents.

Uses: These posses a wide range of solubilities and compatibilities, with make them useful in pharmaceutical and cosmetic preparations.*(27)*

pH value: 6.0

(b) Cremophor RH-40 (BASF, Thailand)

Chemical nature: nonionic solubilizer and emulsifying agent obtained by reacting hydrogenated castor oil with ethylene oxide.

INCI name: PEG-40 Hydrogenated caster oil *(28)*

pH value: 6.0

(c) Ceteareth-20 (Cognis Thai Ltd.)

Chemical nature: nonionic emulsifiers produced by reacting higher saturated fatty alcohols with ethylene oxide.

pH value: 6.0-7.5 *(29)*

(d) Distilled water

The preparation of prophylaxis paste is presented as the flow chart, in Fig 3.4. The compositions of prophylaxis pastes are shown in Table 3.5.

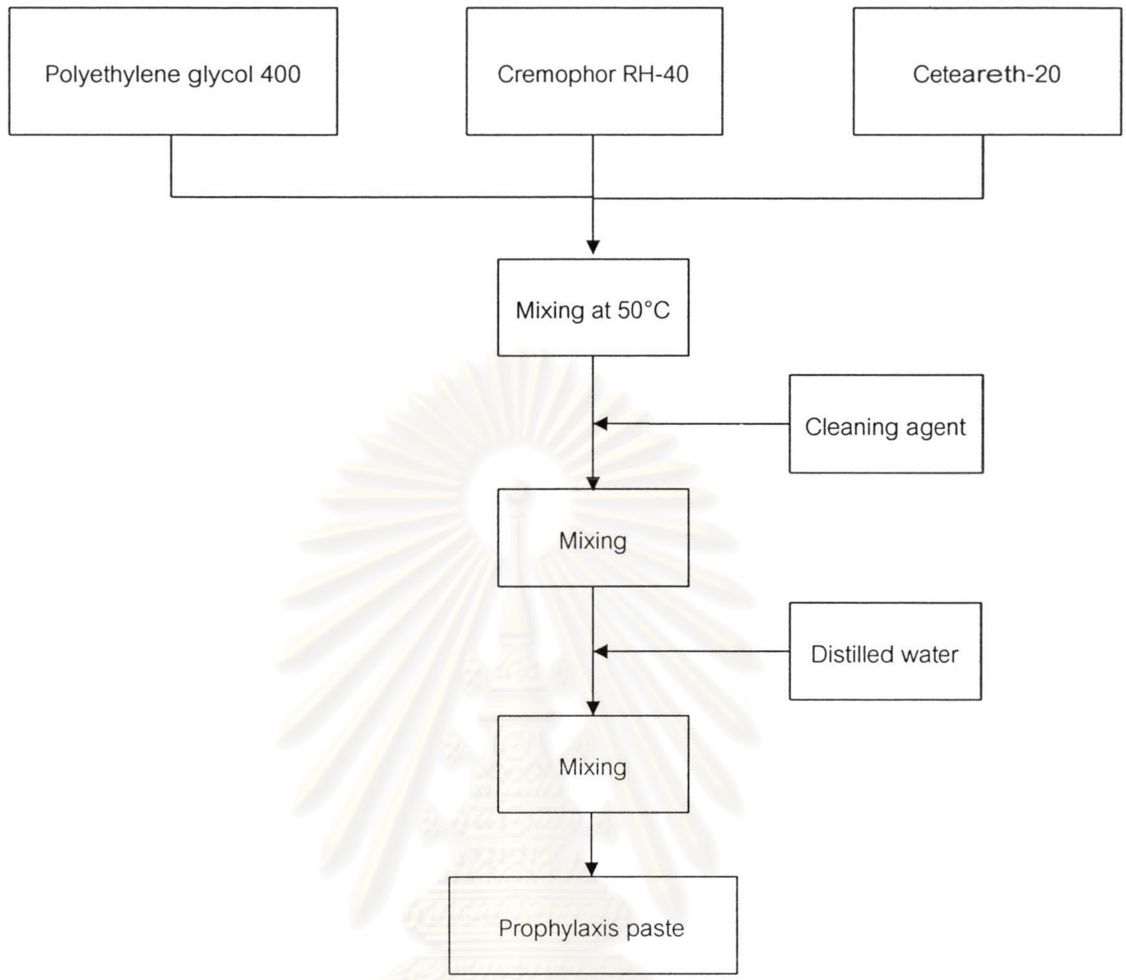

Fig 3.4 Flow chart of prophylaxis paste preparation

Table 3.5 Compositions of prophylaxis paste

	Polishing agent (%)	PEG 400 (%)	Cremophor RH 40 (%)	H_2O (%)	Cetearate 20 (%)	pH*
Nupro						8
Pumice						
106-300 μm	55.53	22.43	15.37	3.77	2.89	6
45-63 μm	60.05	25.47	10.39	4.08	-	6
-16 μm	67.8	20.54	7.04	4.61	-	6
Perlite						
106-300 μm	11.99	58	21.58	3.26	4.99	6
45-63 μm	20.86	38.78	25.99	5.68	8.68	6
-16 μm	47.13	44.75	6.52	1.6	-	6
Diatomite 505						
106-300 μm	35.5	43.33	14.84	6.08	-	8
45-63 μm	37.04	37.41	20.51	5.04	-	8
-16 μm	38.01	47.61	9.207	5.17	-	6
Diatomite hyflow						
45-63 μm	27.8	41.55	23.08	7.56	-	6
-16 μm	36.05	46.61	9.97	7.35	-	6
Calcined diatomite waste						
106-300 μm	39.39	33.42	23.17	4.02	-	7
45-63 μm	42.02	28.86	17.44	5.71	-	7
-16 μm	43.87	42.54	9.108	4.47	-	6
Silica						
106-300 μm	32.36	27.45	11.19	8.8	20.19	7
45-63 μm	37.75	37.75	15.52	5.08	3.89	7
-16 μm	55.72	36.64	5.12	2.52	-	6

Table 3.5 Compositions of prophylaxis paste (continued)

Perlite+Silica (1:1) (Both 106-300 μm)	36.13	40.14	13.75	2.46	7.51	6
Perlite + Silica (1:1) (Both -16 μm)	51.14	20.66	21.23	6.95	-	7
Perlite + Diatomite 505 (1:1) (Both 106-300 μm)	27.47	38.85	15.21	5.6	12.85	7
Perlite + Diatomite 505 (1:1) (Both -16 μm)	46.48	37.55	9.65	6.32	-	6
Perlite + Calcined diatomite waste (1:1) (Both 106-300 μm)	25.18	45.79	29.63	15.41	-	6
Perlite + Calcined diatomite waste (1:1) (Both -16 μm)	35.34	42.83	12.23	9.61	-	6
Diatomite 505 + Silica (1:1) (Both 106-300 μm)	18.86	27.42	22.18	3.85	8.82	7
Diatomite 505 + Silica (1:1) (Both -16 μm)	47.6	32.69	16.47	3.24	-	6
Calcined diatomite waste + Silica (1:1) (Both 106-300 μm)	35.24	29.18	15.85	5.99	13.74	7
Calcined diatomite waste + Silica (1:1) (Both -16 μm)	55.73	27.02	11.57	5.68	-	7

*pH values were used universal indikator pH 0-14

3.5 Characterization and property of prophylaxis paste

3.5.1 The polishing property and surface roughness

The polishing property was assessed on 81 enamel and 81 dentin specimens prepared from freshly extracted human teeth. Their surfaces had been flattened by wet grinding using alumina (0.5-1.5 μm) and diamond paste (0-1μm). Half of the flat surface of each enamel and dentin specimen was covered with a piece of PVC tape, and was regarded as the control side. Then the other side of each enamel and dentin specimen was polished with the prepared polishing paste driven for 30, 60, and 90 seconds with rubber cup at 3,000 rpm and a load of 1.9 N. driven by micromotor (Ultimate 500, Japan) shown in Fig 3.5. During these treatments, the used paste was collected for the study of shape after polishing. The characterization process is schematically showed in Fig 3.6.

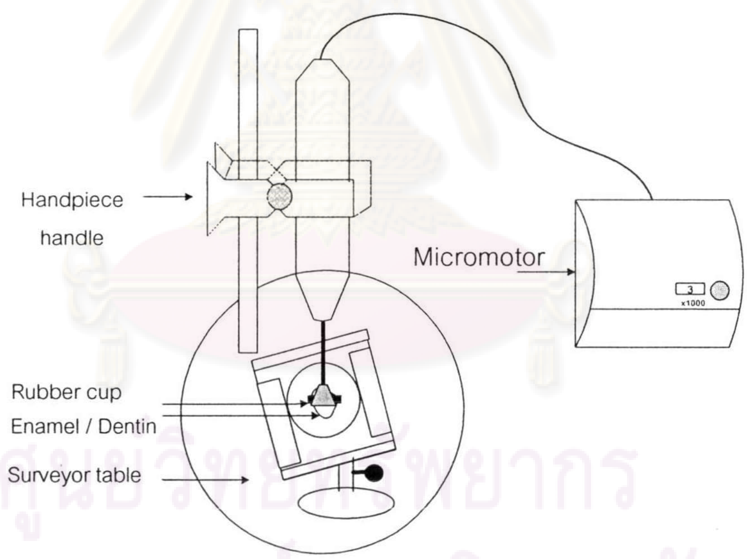

Fig 3.5 Schematic diagram of polishing test

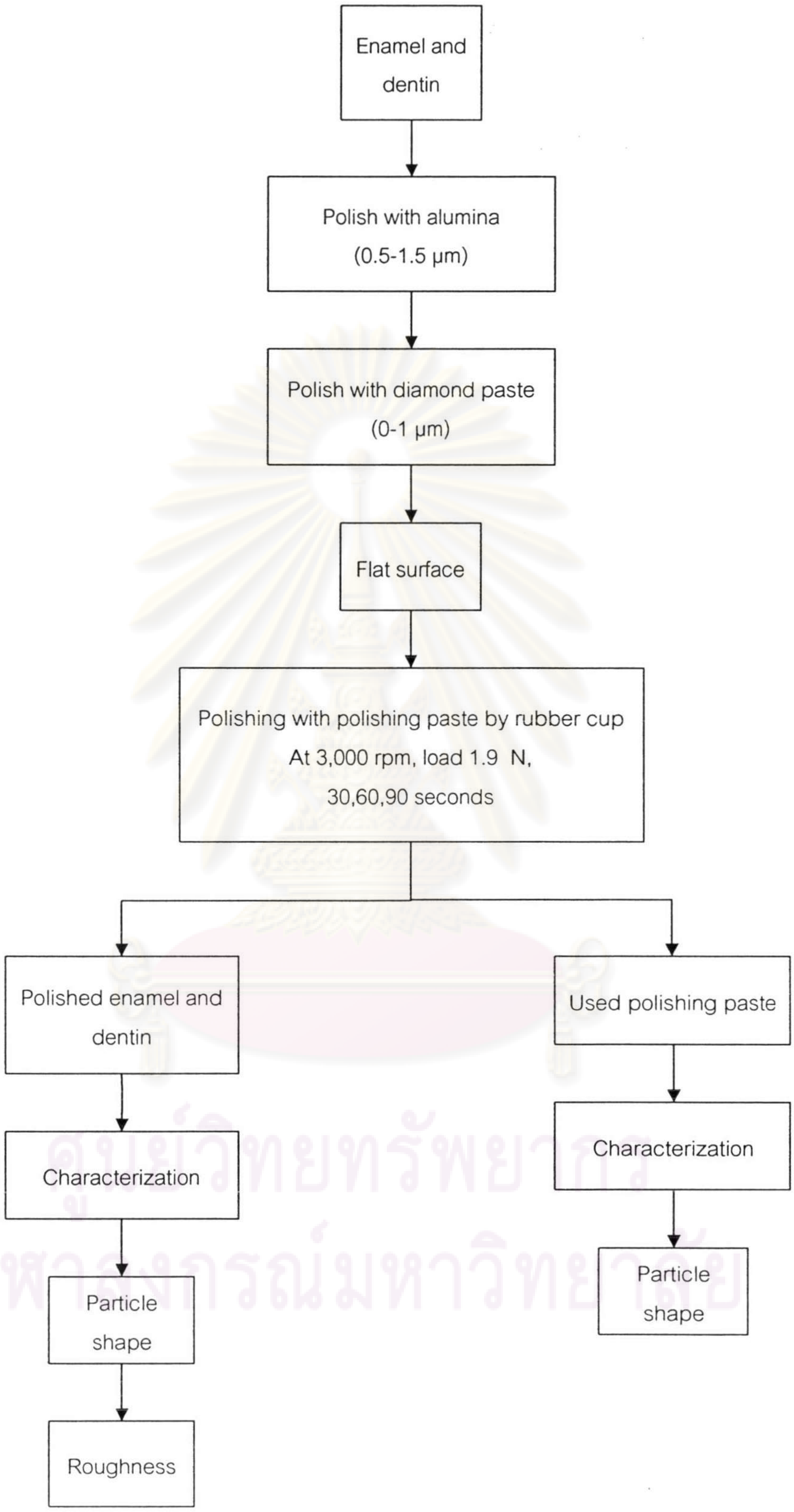

Fig 3.6 Flow chart of enamel and dentin preparation, and polishing paste characterization

The surface roughness induced was recorded as average roughness (Ra in μm) with a profilometer (Taylor Hobson Precision, Talyscan 150). The stylus followed each specimen's surface over a distance of 2.0 mm. at a Gaussian filter, a meter cut-off of 0.25 mm. following ISO 4288-1996.

Average roughness (Ra) is the universally recognized, and most used, international parameter of roughness. It is the arithmetic mean of the absolute departures of the roughness profile from the mean line following the graph in Fig 3.7 and calculated by equation 3.1 *(30)*.

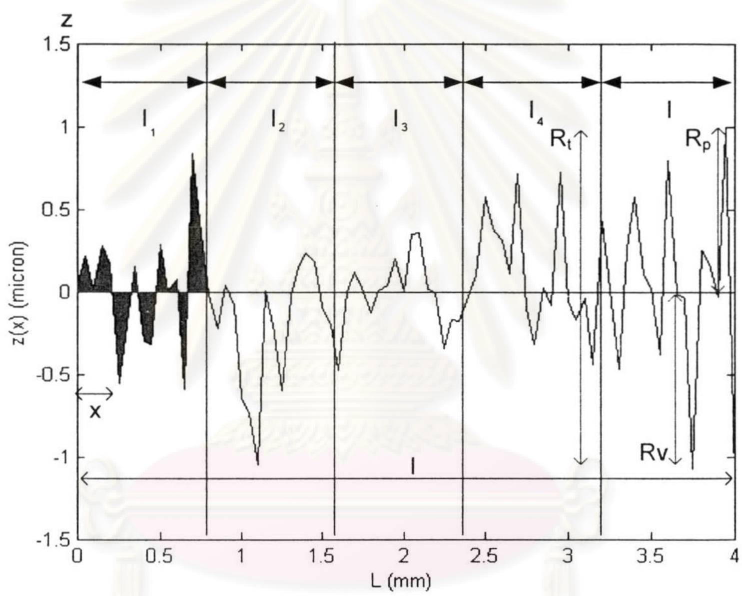

Fig 3.7 The graph for calculation of roughness value

The equation for the calculation of average roughness value is as follows:

$$\text{Ra} = \frac{1}{l}\int_0^l |z(x)|dx \qquad (3.1)$$

X = The cut–off length (mm).

l = The length of profile used for the measurement of the surface roughness parameters.

l , (l_1-l_5) = The sampling length corresponds to filter cut-off length (λ_c).

Rp = The maximum height of the profile above the mean line with in the sampling length.

Rv = The maximum depth of the profile below the mean line with in the sampling length.

Rt = The maximum peak to valley height of the profile in the assessment length.

3.5.2 The morphology of used polishing agent

The shape of used paste was characterized by scanning electron microscope (SEM).

3.5.3 The scratch of the polishing agent on enamel and dentin

The scratches of the polishing agent on enamel and dentin were observed by optical microscope (Olympus BX 60M) at 5X, 10X, 20X, bright field, respectively.