CHAPTER 4

Result and Discussion

4.1 Raw material characterization

Raw materials were characterized for a physical property, chemical property and phase analysis in comparison with control prophylaxis paste.

4.1.1 Chemical composition of raw materials

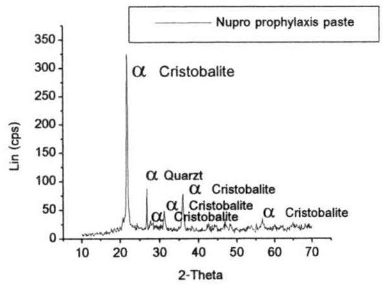
The chemical compositions of raw materials characterized by X-RAY fluorescence spectrometer are shown in Table 4.1.

Table 4.1 Chemical composition of raw materials

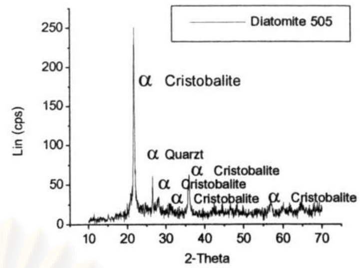
Chemical composition, wt %	Perlite AP-120	Diatomite 505	Diatomite hyflow	Diatomite waste	Silica	Pumice
SiO_2	74.63	87.83	92.93	86.60	98.10	69.94
Al_2O_3	12.70	4.20	3.07	3.73	0.91	12.07
MgO	0.14	0.64	0.52	0.53	0.03	0.22
Na_2O	2.29	4.36	0.36	3.42	0.04	4.29
K_2O	5.86	0.73	0.61	0.67	0.07	4.19
CaO	0.73	0.35	0.31	0.34	0.83	0.97
TiO_2	0.22	0.22	0.17	0.21	-	0.081
Fe_2O_3	1.49	1.57	1.17	1.30	-	1.89

From Table 4.1, as received diatomite hyflow has the highest content of SiO_2 and also the lowest Fe_2O_3. Most of all, SiO_2 is the main composition of all materials. In perlite and pumice that Al_2O_3 content is higher than in diatomite.

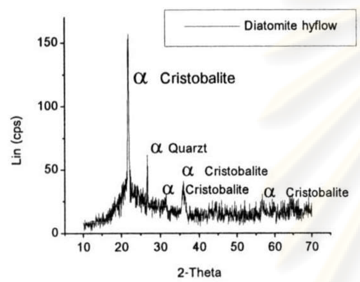
4.1.2 X-RAY diffraction of raw materials



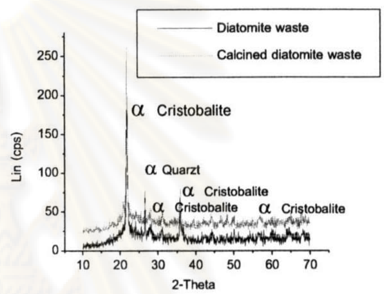
(a) Nupro prophylaxis paste



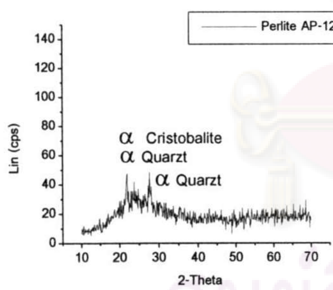
(b) Diatomite 505



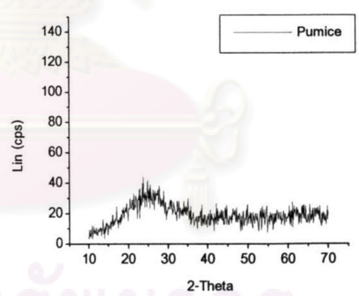
(c) Ditomite hyflow



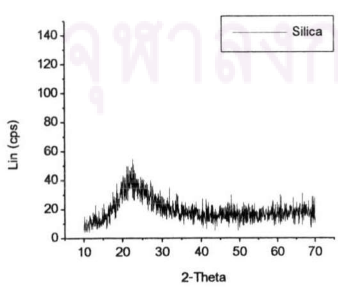
(d) Diatomite waste



(e) Perlite AP-120



(f) Pumice

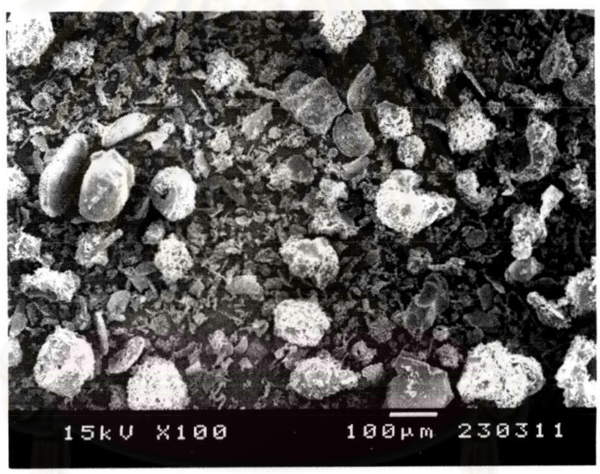


(g) Silica

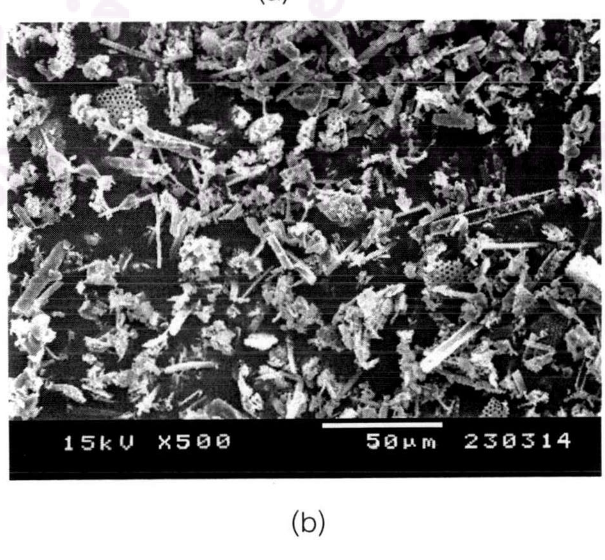
Fig 4.1 XRD patterns of raw materials

From the x-ray diffraction patterns of raw materials in Fig 4.1, Nupro prophylaxis paste (a) consists of mainly α-cristobalite at 2-theta 21.77 and some α-quartz at 26.62 and some amorphous phase similar to diatomite 505, diatomite hyflow and diatomite waste therefore it is presumed that the composition of Nupro prophylaxis paste is diatomite. For perlite AP-120, pumice and silica in Fig 4.1 (e), (f), (g) consist mostly of amorphous phase. However, there is also some small amount of crystalline material in perlite, diatomite waste and calcined diatomite waste, from XRD pattern. The phase compositions of both diatomite wastes before and after calcining at 400°C were similar.

4.1.3 Particle size and shape of raw materials

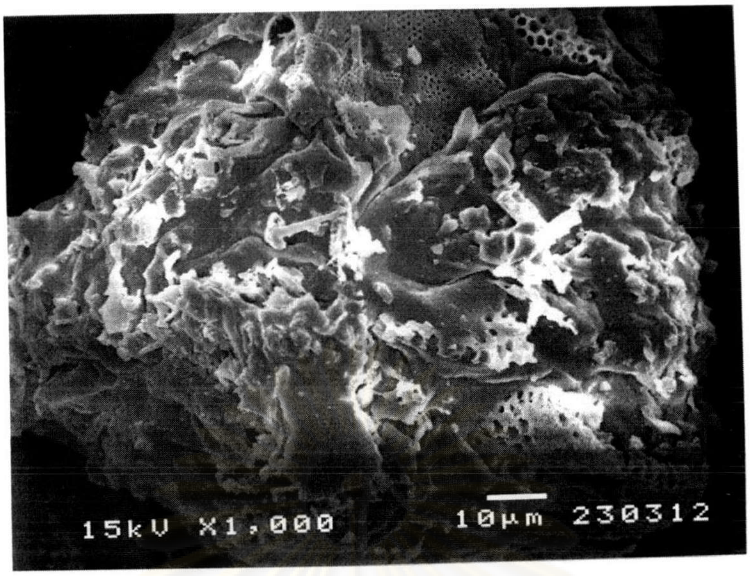


(a)



(b)

Fig 4.2 SEM micrographs of diatomite 505



(c)

(d)

Fig 4.2 SEM micrographs of diatomite 505 (Continued)

(a) Diatomite 505

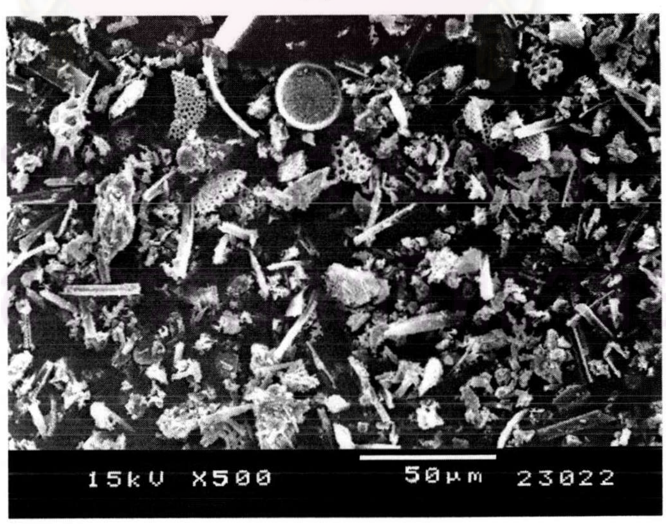
(b) Diatomite 505, particle size -16 μm

(c) Agglomerate of diatomite

(d) Rod shape of diatomite particle

From the SEM micrographs of diatomite 505, in Fig 4.2 (a) the particle shapes are spherical spongy, porous shapes which are classified as formed-convex surface particles appearing boulder-like. Most of diatomite -16 μm, Fig 4.2 (b) is composed of rod shape and fragmented, honeycomb-like particles. Hence, they were classified as favosed particles regarding honey comb-like sheet, and as fragmented regarding ragged asymmetrical particles *(15)*. However, it appears to be a mixture of all the mentioned shapes in the two size diatomite.

(a)



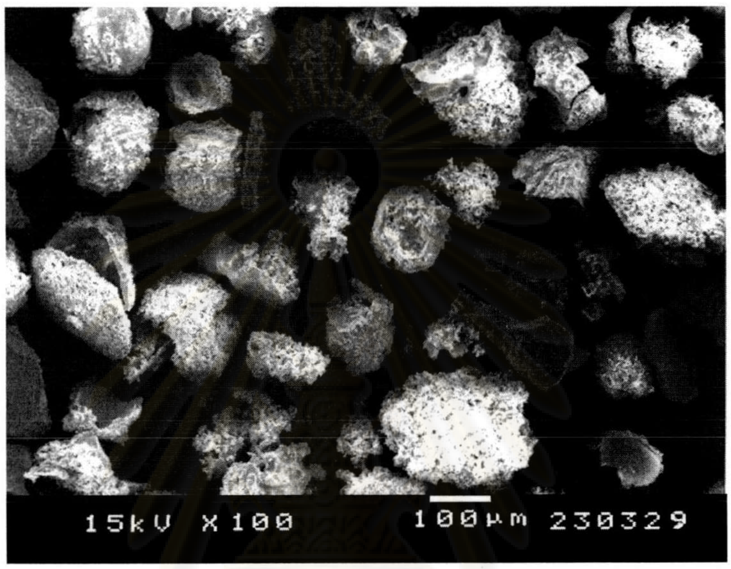
(b)

Fig 4.3 SEM micrographs of diatomite hyflow

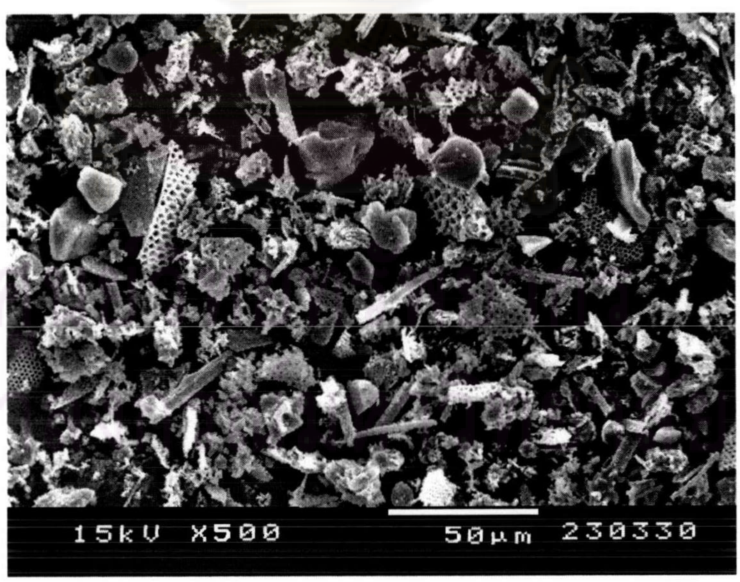
(a) Diatomite hyflow, particle size 45-63 μm

(b) Diatomite hyflow, particle size -16 μm

For particle shape of diatomite hyflow, 45-63 μm, from Fig 4.3 (a) the particles are composed of wheel shapes showing characteristic honeycomb-like. From Fig 4.3 (b) diatomite hyflow, particle size -16 μm, shows the fragmented honeycomb-like structure of favosed type.



(a)



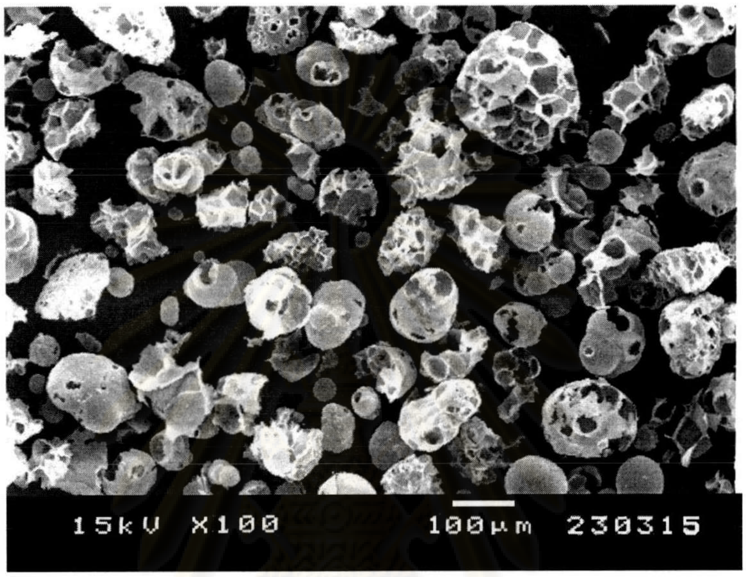
(b)

Fig 4.4 SEM micrographs of diatomite waste

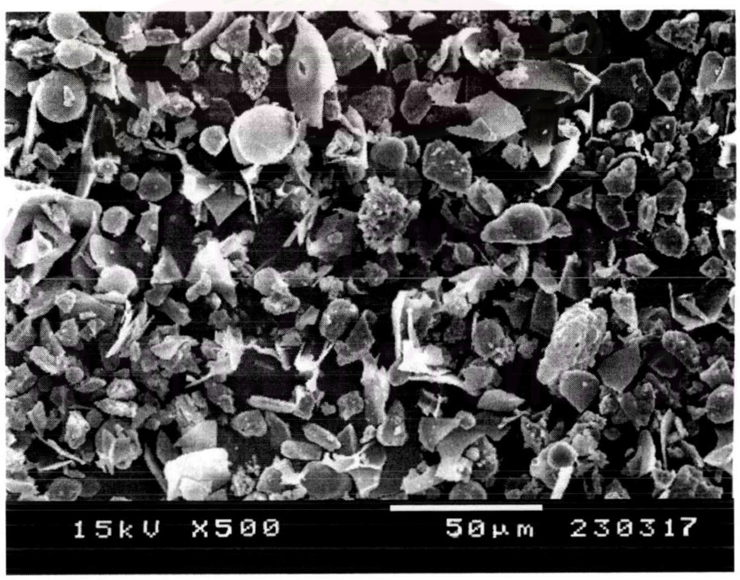
(a) Diatomite waste, particle size 106-300 μm

(b) Diatomite waste, particle size -16 μm

From Fig 4.4 (a) The particles of diatomite waste, 106-300 μm, are spherical spongy, porous shape and boulder-like. Fig 4.4 (b), particle size -16 μm, shows the fragmented honeycomb-like structure and is also favosed.

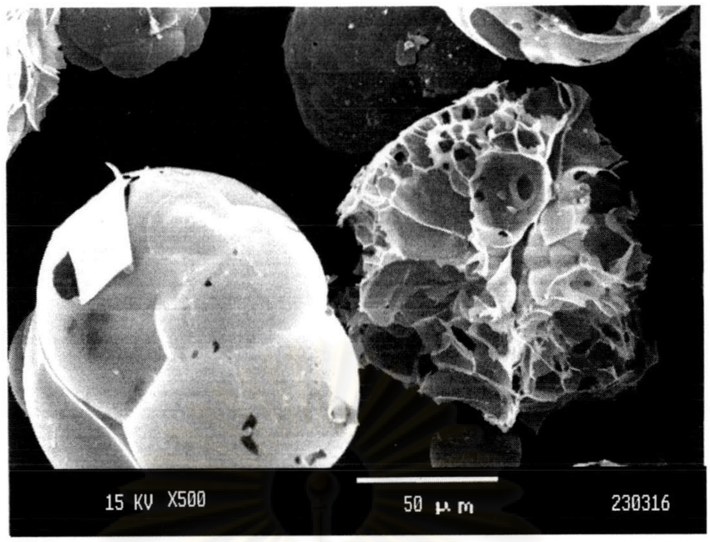


(a)



(b)

Fig 4.5 SEM micrographs of perlite



(c)

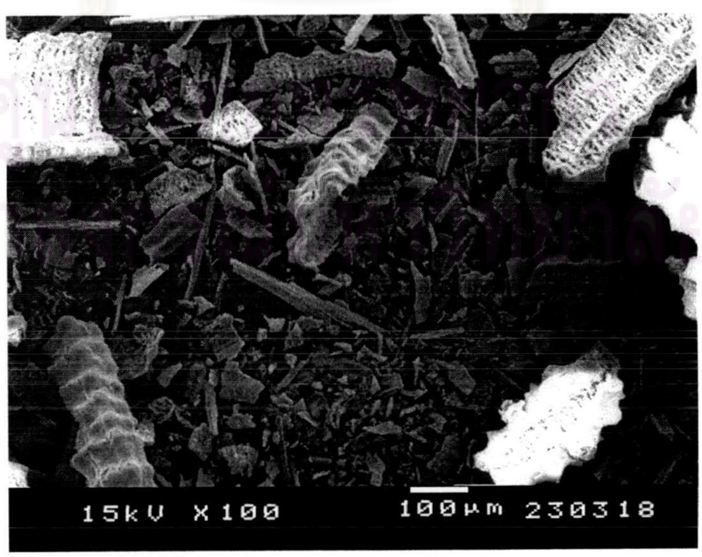
Fig 4.5 SEM micrographs of perlite (Continued)

(a) Perlite, particle size 106-300 μm

(b) Perlite, particle size -16 μm

(c) Solid part of a grain of perlite in (a) enlarged

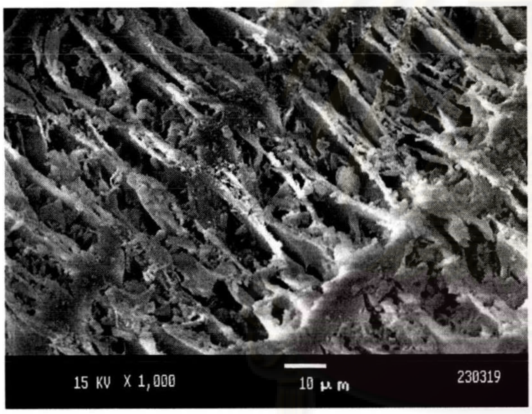
The particles of perlite size 106-300 μm, from the SEM micrograph of Fig 4.5 (a), are round and contain of pore inside like popcorn, hence the shape is also spherical, and porous. Fig 4.5 (b), particle size -16 μm, shows flat sheet-like or flakes from fragments of large particles.



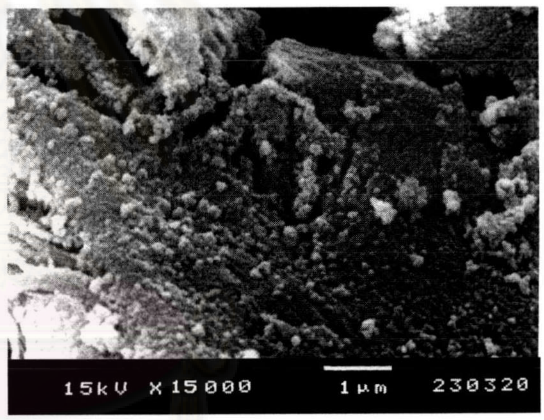
(a)

Fig 4.6 SEM micrographs of silica

(b)



(c)



(d)

Fig 4.6 SEM micrographs of silica (Continued)

(a) Calcined silica from rice husk

(b) Silica particle size 106-300 μm

(c), (d) Solid part of a grain of silica in (a) enlarged

The particle shape of calcined silica in Fig 4.6 (a) is sharp, lathe-like shape *(31)*. Fig 4.6 (b), the shape of silica particle size 106-300 μm, is similar to calcined silica. Fig 4.6 (c), shows the surface of silica grain. Fig 4.6 (d) displays the conglomerate mass of fine spherical particles on the large surface of silica grain.

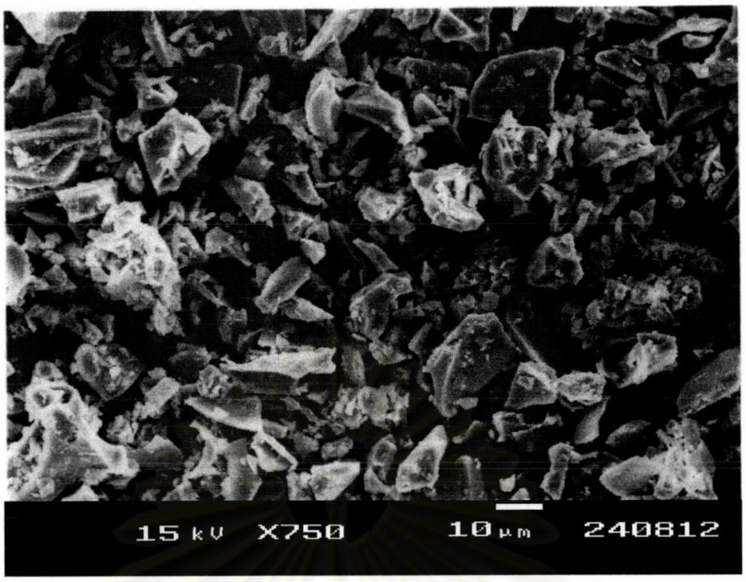
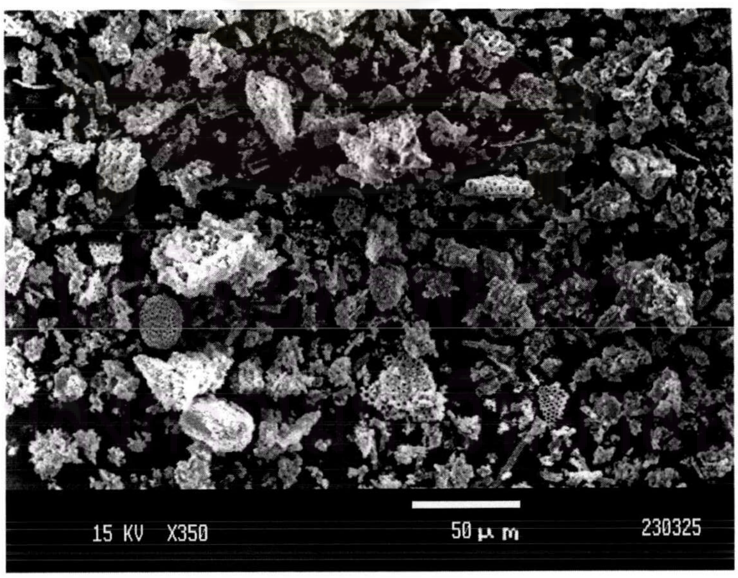


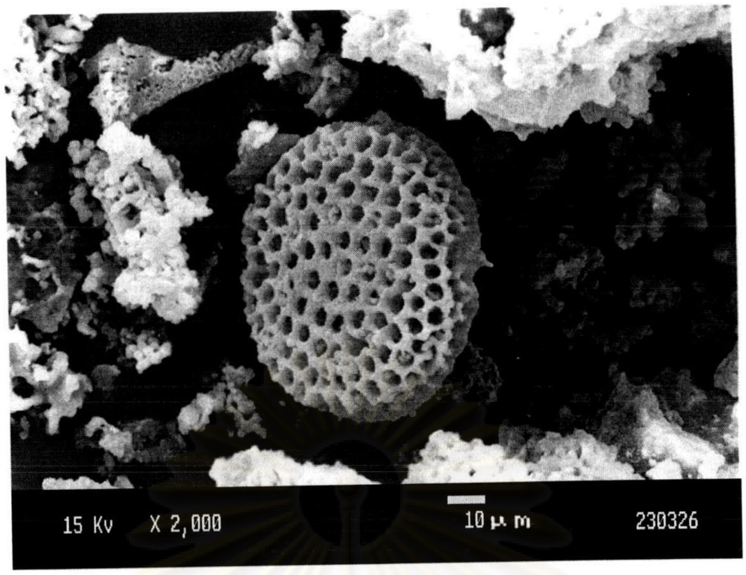
Fig 4.7 SEM micrograph of pumice

From the micrograph of Fig 4.7 the particles of pumice show granular, irregular shape.



(a)

Fig 4.8 SEM micrographs of Nupro prophylaxis paste



(b)

Fig 4.8 SEM micrographs of Nupro prophylaxis paste (Continued)

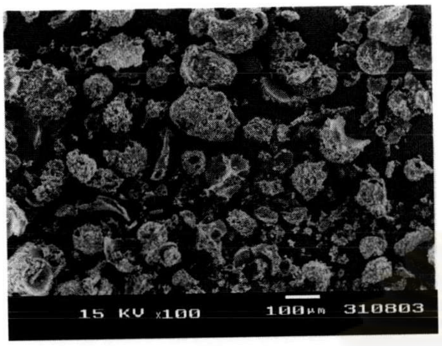
(a) Nupro prophylaxis paste

(b) Solid part of a grain of silica in (a) enlarged Nupro prophylaxis paste

From Fig 4.8 particles of Nupro show the mixed appearance of diatomite 505 and hyflow. Furthermore the particles from the micrograph consist of two basic sizes. The larger particles are polisher and smaller particles are cleaner, randomly.

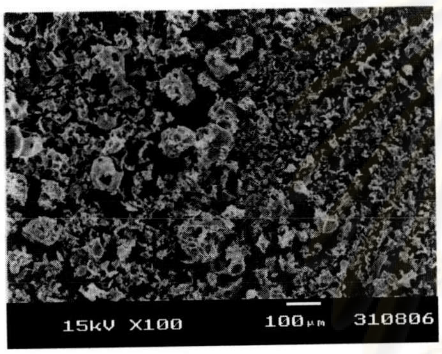
4.2 Characterization of prophylaxis paste

4.2.1 SEM micrographs of used polishing agents

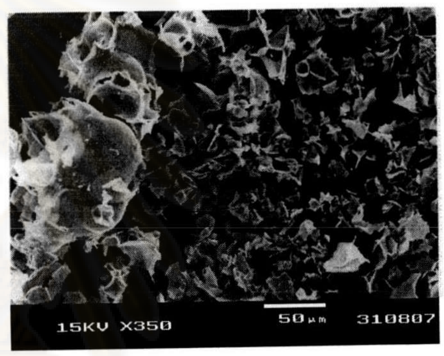


(a) Diatomite 505, 106-300 µm

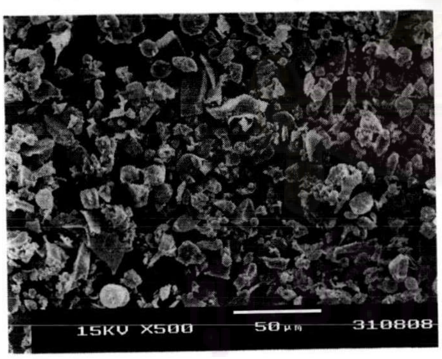
(b) Diatomite hyflow, -16 µm



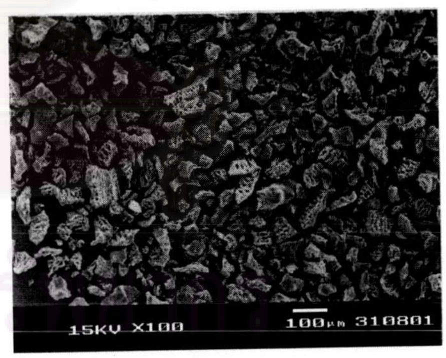
(c) Perlite, 106-300 µm



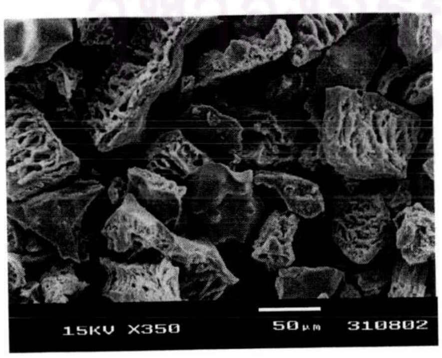
(d) Enlarge of perlite, 106-300 µm



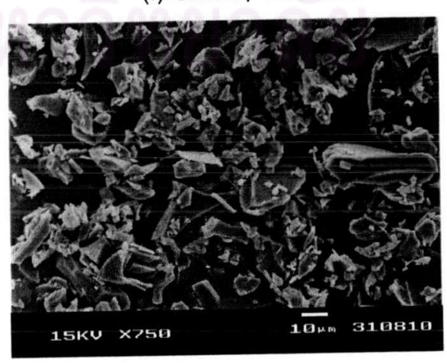
(e) Perlite, -16 µm



(f) Silica, 106-300

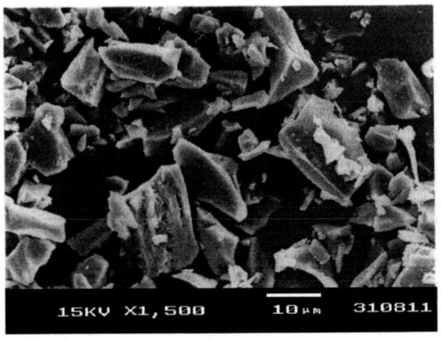


(g) Enlarge of silica, 106-300 µm

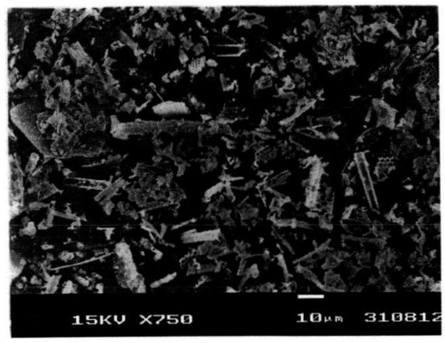


(h) Pumice, -16 µm

Fig 4.9 SEM micrographs of used polishing agents



(i) Enlarge of pumice, -16 µm



(j) Nupro

Fig 4.9 SEM micrographs of used polishing agents (Continued)

From the SEM micrographs of some of the used prophylaxis agents, Fig 4.8, the polishing agent becomes rounded in shape and sharp edges become blunt. In addition, from Table 4.2 the particle size is decreased from the unused prophylaxis paste because the particle abrades the dentin or the enamel surface and small amounts of particle mass is transferred, or as a result of being fragmented.

Table 4.2 Average particle size of used polishing agent

Polishing agent	Average particle size (µm)
Nupro, 22.22 µm	10.75
Diatomite 505,103.44 µm	94.32
Diatomite hyflow,13.51 µm	10.25
Perlite, 97.41 µm	50.47
Perlite, 12.90 µm	10.55
Silica, 75.16 µm	70.85
Pumice, 16.26 µm	11.98

4.2.2 Surface roughness of enamel and dentin

Table 4.3 Surface roughness of enamel and dentin

Gr.	Polishing agent	Ra (μm), Dentin 30 s			Ra (μm), Enamel 30 s		
		Flat surface	After polishing	ΔRa	Flat surface	After polishing	ΔRa
1	Nupro	0.256	0.138	-0.024	0.110	0.079	0.035
2	Silica, 106-300 μm	0.054	0.152	-0.038	0.063	0.053	0.061
3	Silica, 45-63 μm	0.083	0.091	0.022	0.048	0.044	0.069
4	Silica, -16 μm	0.098	0.081	0.033	0.030	0.181	-0.067
5	Diatomite 505, 106-300 μm	0.080	0.106	0.008	0.077	0.094	0.020
6	Diatomite 505, 45-63 μm	0.160	0.018	0.096	0.055	0.069	0.045
7	Diatomite waste, 45-63 μm	0.037	0.054	0.060	0.104	0.099	0.015
8	Diatomite hyflow, -16 μm	0.053	0.052	0.062	0.024	0.047	0.067
9	Perlite AP-120, 106-300 μm	0.091	0.141	-0.027	0.116	0.097	0.017
10	Perlite AP-120, 45-63 μm	0.094	0.112	0.002	0.129	0.137	-0.023
11	Perlite AP-120, -16 μm	0.131	0.095	0.019	0.109	0.072	0.042
12	Perlite AP-120+Silica (1:1) (Both 106-300 μm)	0.110	0.083	0.031	0.272	0.325	-0.211
13	Perlite AP-120+Silica (1:1) (Both -16 μm)	0.288	0.176	-0.062	0.122	0.262	-0.148
14	Perlite AP-120+Diatomite 505, (1:1) (Both 106-300 μm)	0.144	0.052	0.062	0.073	0.134	-0.020
15	Perlite AP-120+Diatomite 505 (1:1) (Both -16 μm)	0.096	0.050	0.063	0.298	0.081	0.032
16	Diatomite 505+Silica (1:1) (Both 106-300 μm)	0.117	0.160	-0.046	0.381	0.255	-0.141
17	Diatomite 505+Silica (1:1) (Both -16 μm)	0.081	0.062	0.052	0.076	0.071	0.043
18	Pumice, 106-300 μm	0.056	0.088	0.026	0.075	0.223	-0.109
19	Pumice, 45-63 μm	0.071	0.087	0.026	0.062	0.136	-0.022
20	Pumice, -16 μm	0.175	0.158	-0.044	0.054	0.084	0.030
	Average Ra of flat surfaces	0.114			0.114		

For dentin and enamel after polishing or grinding with prepared prophylaxis paste their surface can be described in term of average roughness (Ra). Since the flat surfaces of both dentin and enamel have to be prepared by grinding with alumina powder (Al_2O_3), (0.5-1.5 μm) and polishing with diamond paste (0-1 μm) prior to polishing with the prepared prophylaxis paste. The average roughness (Ra) value of the flat surface is later taken as the control in comparison with the Ra after polishing with prophylaxis paste.

If Ra after polishing is smaller than Ra flat surface, it means that some of mass of dentin or enamel is transported and result in smooth surface because there are more small and shallow cuts than the deep ones. From the average roughness graph in Fig 3.7 the height between peak and valley (R_t) is reduced hence results in the decrease in the average roughness.

If the average roughness is large, it means that the mass is transported, less small and shallow cuts, than bigger and/or deeper ones. The height between peak and valley (R_t) increases.

To consider the effects between of small grains and large grains or shape of polishing particles on dentin and enamel. The natural phenomena of friction and wear between the two moving objects must be briefly discussed.

The 3 laws of friction *(32)*, generally known as Amonton's law can be stated as follows:

1. The frictional forces is proportional to the load W.
2. The force is independent of the geometric area of contact between the two objects.
3. The kinetic frictional force for a system is approximately 1/3 of the value of the normal load.

The first and generally most important is that of adhesion between points of actual contact between the surfaces. The true area of contact between two unlubricated surfaces will be less than the apparent area. At those areas of contact, the two surfaces will be bound by a certain adhesion force arising from the interaction between the materials at the molecular level. For the two surfaces to move tangentially, the points of adhesion must be sheared or broken. If the area of contact is A, the shear strength of the bond is s, then the friction force due to adhesion will be

$$F_{ad} = As \tag{4.1}$$

The second parameter contributes to total friction (F) is deformation (F_{def}), and F may be written as

$$F = F_{ad} + F_{def} = As+P \tag{4.2}$$

Without going into detail about the exact process involved the F_{def} is replaced by the non specific term P.

If a normal force, W is applied to the system (In the experiment it was 0.6 N.), it is expected that the added pressure on the asperities will cause some deformation leading to an increase in the real area of contact *(32).*

If the materials respond to the added pressure by plastic deformation (i.e., a permanent change in the shape of the asperities brought about by the application of the mechanical force), the real area of content can be written as

$$A = \frac{W}{T_0} \tag{4.3}$$

Where T_0 is the yield strength of the band, for two objects of the same material, the coefficient of friction, μ_f can be defined as

$$\mu_f = \frac{F}{W} = \frac{S}{T_0} \tag{4.4}$$

Crystals with relatively isotropic structures like diamond sapphire have $\mu_f \sim 0.1$. Anisotropic or layered crystalline materials such as graphite and talc are known as good lubricants (lower friction) and also have low μ_f (~0.1). In summary, materials with low μ_f will deform elastically rather than plastically. However, beyond a certain load, they also show surface damage with increased μ_f value.

If the two surfaces are of different materials, with different hardness, asperities on the hard surface will tend to plow into the soft surface, forming grooves.

The total coefficient of friction will be a combination of adhesion and plowing contributions. If the asperities on a surface are known to be relatively blunt, it can assumed that they will not make a significant contribution in terms of the plowing mechanism.

Before discussing on the polishing test results, it is very important to mention about the compounding of the experimental prophylaxis pastes due to the difference in the nature of each polishing agent employed, i.e. shape, particle size, porosity, mineral type, etc. To compound each of the solid into a paste of required consistency is complicated since large, porous particles (low bulk density) highly absorb solution hence need more solution to accommodate their large volume, resulting in low density paste. On the contrary, fine particles(high bulk density) can easily be accommodated into high density paste. For this reason, in Table 3.5 the solid contents of the prepared pastes widely vary. The lowest is paste of perlite with large particles,12 wt%, to the highest , 68 wt% of paste of pumice with fine particles. Therefore, with no regard to the kind of solid, there is a wide distribution between solid content among the pastes, with fine particles (35-68 wt%), with medium particles (21-60 wt%) and with large particles (12-56 wt%). It is noted that all the pastes of pumice contain high solid content, 56-68 wt% and those of perlite contain the lowest, 12-47 wt%.

The results of Ra in Table 4.3 are graphically presented in Fig 4.10 and Fig 4.11

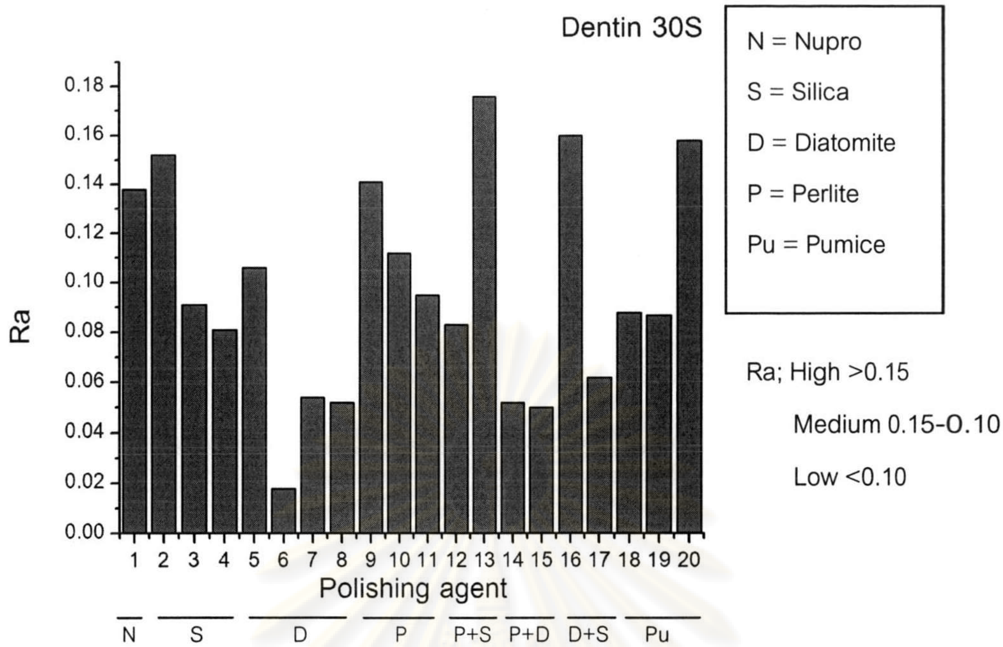


Fig 4.10 Average roughness of dentin after polishing

From Fig. 4.10 and Table 4.3, it is seen that the Ra values after polishing test on dentin with the prepared prophylaxis pastes range from 0.052-0.176. The prophylaxis pastes that show low Ra values are better polishes. Pastes containing silica show low to medium Ra values in the following order: paste with large particles > paste with medium particles > paste with fine particles. Pastes containing diatomite show low to medium Ra values, and the trend is the same as those containing silica. Pastes containing perlite show low to medium Ra values and their Ra values also decrease with decreasing particle size. Pastes containing pumice show low to high Ra values. However, the paste with fine pumice particles shows a quite high Ra value due to its very high solid content. For pastes with mixed polishing agents: Ra values of paste containing perlite + silica are from low to high. Ra value of paste with large particles (36 wt %), is less than that of paste with fine particles (51 wt%). This is an obvious effect of solid content. Ra values of pastes containing perlite + diatomite are low, and Ra values of pastes with either large or fine particles are approximately the same. This, to a certain extent, is due to the less abrasive nature of diatomite. Ra values of pastes containing diatomite + silica are from low to high and in the normal trend. Nupro shows medium Ra value corresponding to its constituent, diatomite.

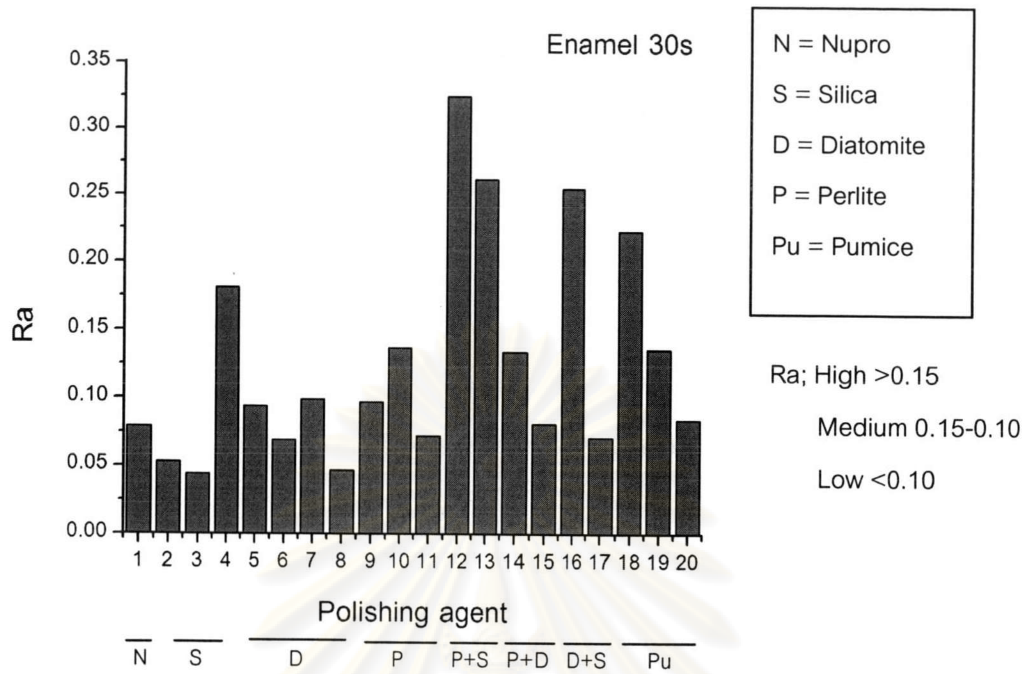


Fig 4.11 Average roughness of enamel after polishing

Fig. 4.11, the polishing test of prophylaxis pastes on enamel shows the Ra values after polishing ranging from 0.044-0.325. Pastes containing silica show very low to medium Ra values. The Ra values tend to decrease with decreasing particles with the exception of paste with fine particles which effected by the high solid content. Pastes containing diatomite show low Ra values. Pastes containing perlite also show low to medium Ra values, and the low Ra value of paste with large particles is due to the very low solid content which may not be sufficient for polishing. Pastes containing pumice show low to high Ra values in the normal order of particle size.

For pastes with mixed polishing agents, Ra values of pastes containing either large or fine particles of perlite + silica show very high Ra values which may be due to the effect of large particles plus high solid content. Pastes containing perlite + diatomite show low to medium Ra values with normal trend. Pastes containing diatomite + silica show low to high Ra value, also with normal trend. Nupro paste shows quite a low Ra value, and is competitive with pastes containing either silica or diatomite or perlite. However, it is quite complicated in making decision on comparison since there are complications regarding the nature of different materials and solid content in the compounding of the paste. The general trend is that pastes with large particle size should show high Ra values. The hardness of enamel (~5-8 Mohs) is much higher than that of dentin (~1-2 Mohs). Then the good polishes should have competitive hardness and it is also believed that materials with very fine particles. I.e. fume silica, amorphous alumina, etc. should exhibit lower hardness than when they are present as bigger particles.

The test on the effect of polishing time on Ra values

The results of Ra values of both dentin and enamel specimens obtained after polishing with selected prophylaxis pastes at 30, 60 and 90 seconds are plotted against polishing time, in Fig. 1 and 2 and attached in the Appendix C. There is no correlation between Ra values and polishing time found in this experiment. But there is a trend that the Ra values of most of the pastes increase with time, but after 60 seconds they tend to decrease, which indicates the breaking and blunting of the particles. However, the Ra value from paste with large or coarse particles of perlite seems increase further from 60 seconds. This is probably due to the incomplete breaking of its large particles. It is noted that pastes containing silica, either coarse or fine particles, are good polishes for enamel in the whole range of the experimental

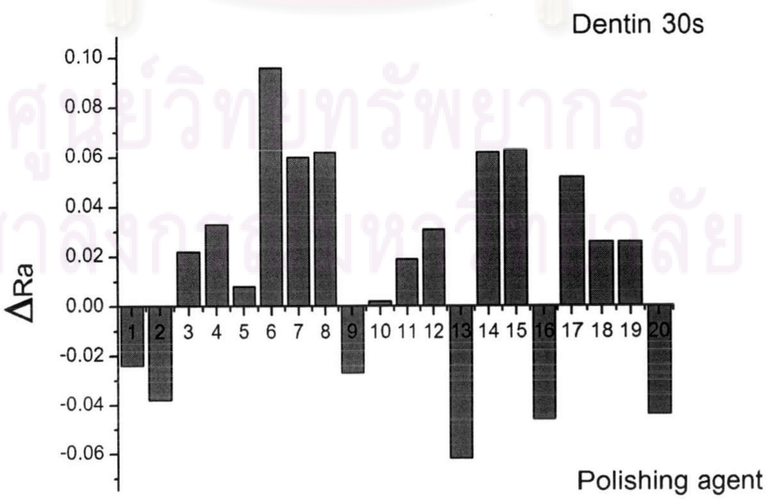


Fig 4.12 Average roughness of dentin between flat and polished surface

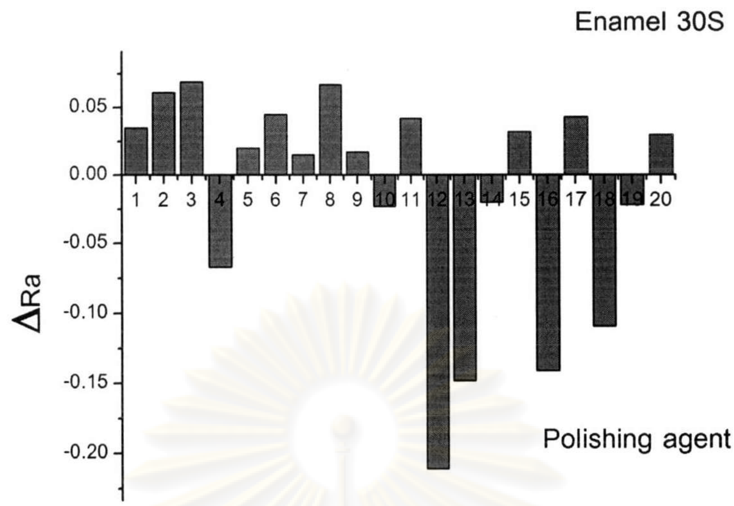


Fig 4.13 Average roughness of enamel between flat and polished surface

However, the values of Ra_{after} so far discussed, can just tell how smooth or how rough the polished surface is. They can not accurately tell which paste is a better polish since the polishing test of all the experimental pastes has not individually performed on the same flat surface of the same tooth specimen, due to the small size of the tooth sample: It is obvious that the nature of human teeth can greatly vary from one to another. Therefore, even using the same condition in the preparation of flat surface on each half of a tooth, it is difficult to get a narrow range of the Ra values of both dentin and enamel. The ranges of Ra values of the flat surface of dentin and enamel are from 0.037-0.288 and 0.024-0.351, respectively. Accordingly the Ra values of the flat surface must be normalized to be only one value which is the average Ra value of all the tests (20 specimens). As seen in Table 4.3, it is found that the average Ra value of both 20 dentin and 20 enamel specimens happens to be the same value, 0.114. In order to compare the performance of the experimental prophylaxis pastes, ΔRa is deduced from Ra_{flat}- Ra_{after} of each test paste, and the graphic illustration is shown in Fig 4.12 and 4.13.

It is seen from Fig 4.12 that about half of the prophylaxis pastes have the ΔRa values in the positive side which means that they have lower or smaller Ra values (smoother surface) than the normalized flat surface of dentin, and the rest (including Nupro) has larger Ra, hence half of the pastes are good polishes. Considering only positive ΔRa the trend is that the ΔRa values of diatomite> silica> perlite.

Pastes from large particle diatomite and medium particle perlite show small ΔRa, and the large ΔRa values are from the diatomite paste with fine particles, and from the mixture of diatomite and perlite, both with fine and coarse particles. The graphic illustration of Fig 4.13 shows the test on enamel. The ΔRa values of pastes from silica > diatomite > perlite. This may explain that materials with higher hardness are needed for enamel. However the trend of the effect of particle size on dentin and enamel has to be considered together with its shape and solid content. From the results it is reasonable to suggest that, for both dentin and enamel, medium to fine fragmented particles seem to show large ΔRa, and hence pastes with fine particles are better polishes.

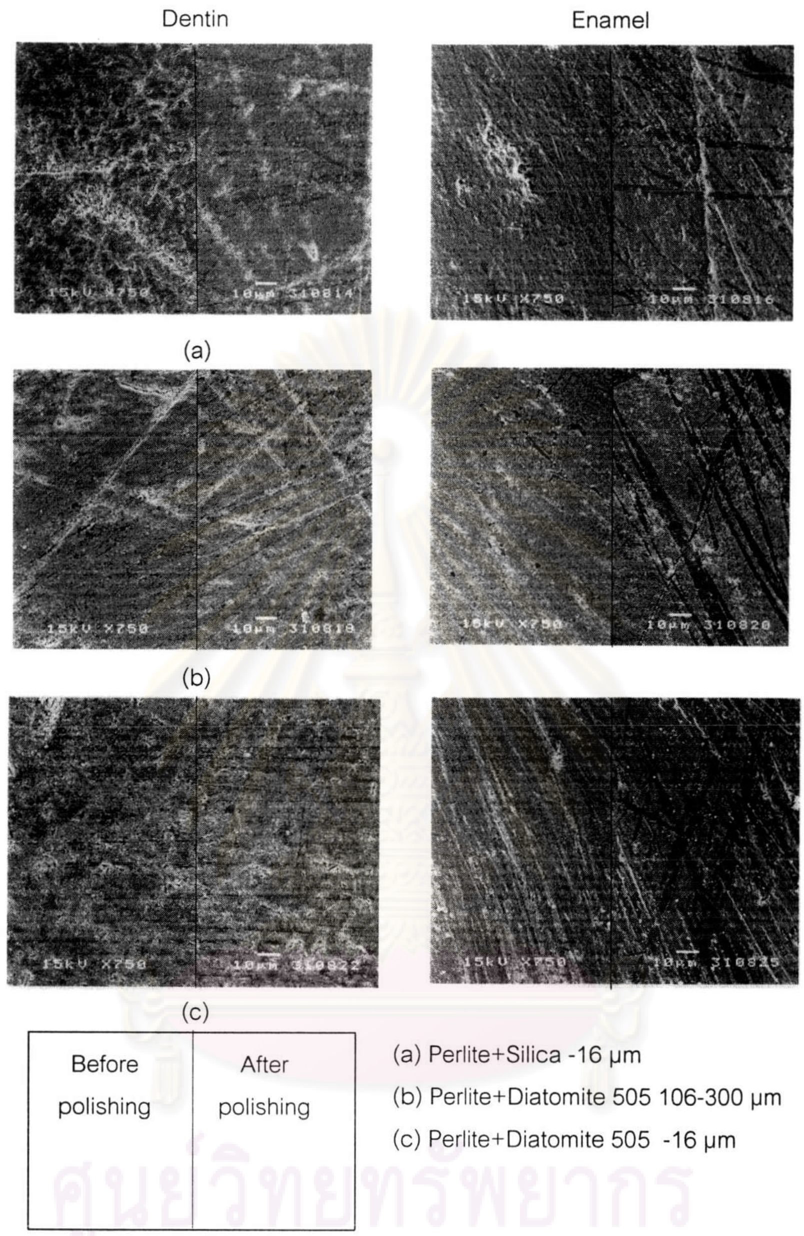


Fig 4.14 SEM micrographs of dentin and enamel polished with mixing polishing agents.

Fig 4.14 shows the SEM micrographs of the flat and polished surfaces of dentin and enamel in comparison. For dentin, surfaces in (a) is rougher than the flat surface while those of (b) and (c) are smoother than their corresponding flat surfaces. For enamel, surfaces in (a) and (b) are rougher than the corresponding flat surfaces but surface in (c) is smoother.