CHAPTER IV

EXPERIMENTAL

4.1. Cellulose nitrate solution

Cellulose nitrate resin as received was treated at 100°C for 1 hour in the oven as shown in Figure 4.1 to remove isopropyl alcohol and moisture then took up water to adjust the water content of cellulose nitrate powder to be 1, 2, 3, 4, 6, 8, and 10 % by weight, the Karl Fisher titrator in Figure 4.2 was used to checked water content in cellulose nitrate resin. Cellulose nitrate solution obtained by mixing 10 gram the CN powder at various water contents using a mixed solvent system as follows: ethyl alcohol 5 gram, butyl acetate 15 gram, ethyl acetate 20 gram, and toluene 45 gram. To study an effect of water content in the cellulose nitrate solution on nail enamel's properties, the characteristics of the solution were examined including viscosity, and drying time, and film properties such as adhesion, gloss, hardness, and surface texture.

Figure 4.1 Oven.

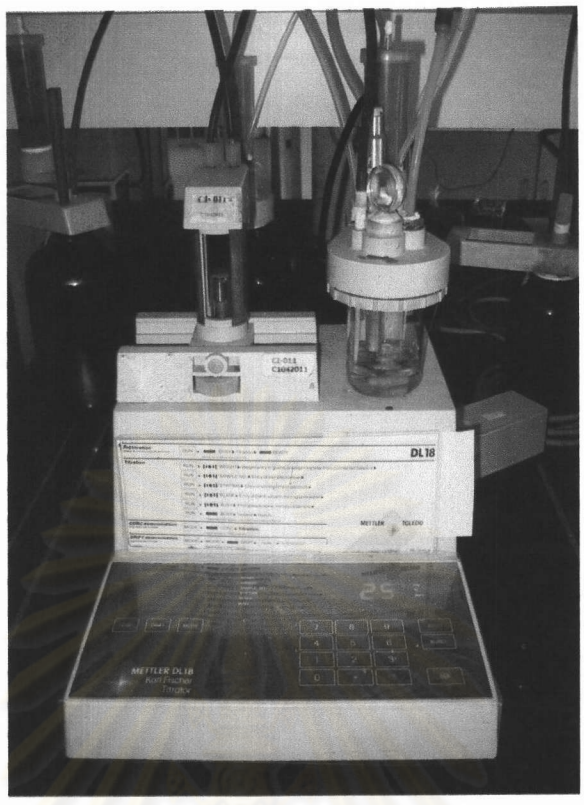


Figure 4.2 Karl Fisher titrator.

4.1.1 Flow behavior

Cellulose nitrate solution at various content were measured flow behavior using a rotational viscometer, Physica Rheolab MC1: Standard measuring system MS-Z3 DIN/MC1 as shown in Figure 4.3 and 4.4, at shear rate 30-1000 s^{-1} for 10 minutes. The volume of measured solution was about 17-20 gram. The raw data was plotted shear rate versus viscosity of the solutions and shear stress.

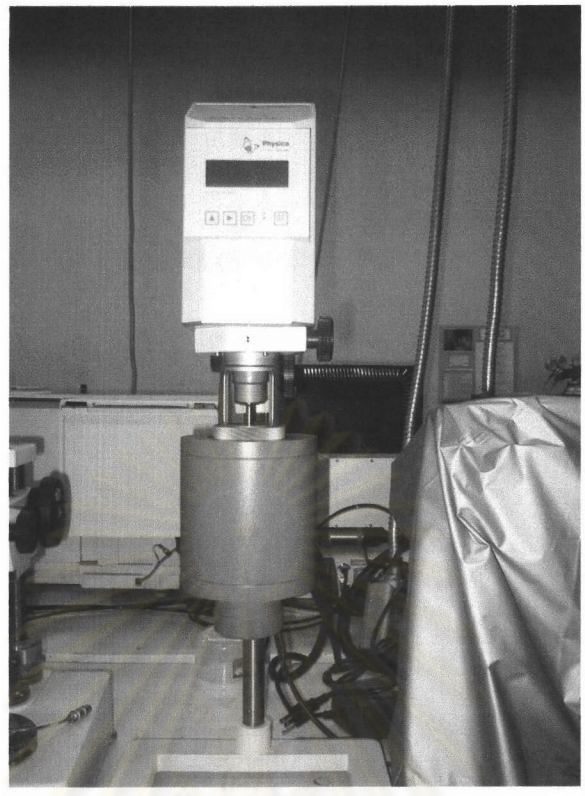


Figure 4.3 Viscometer.

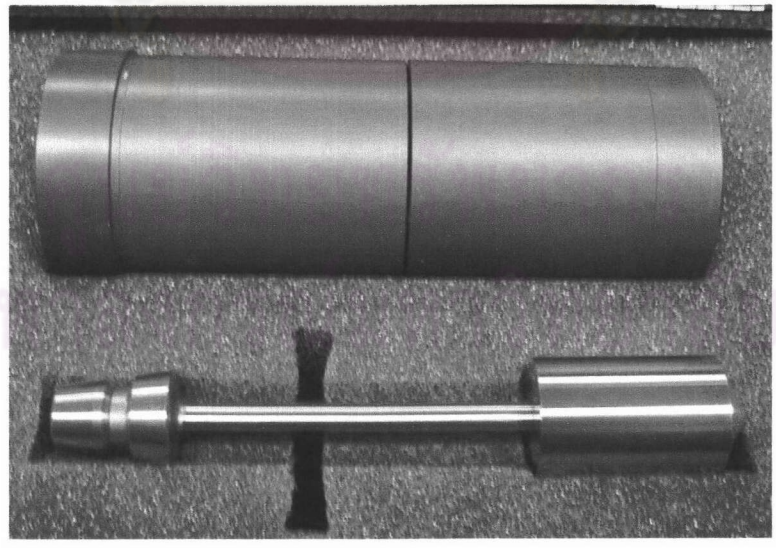


Figure 4.4 Coaxial standard measuring system MS-Z3.

4.1.2 Drying time

Drying time of cellulose nitrate solution was conducted following ASTM 1640 at temperature 25 °C and 50±5 % relative humidity in the humidifier control room as shown in Figure 4.5. All test specimens were prepared and tested by one operator applying 10 gram of various water content cellulose nitrate solution on the clean glass panels and casting by applicator as shown in Figure 4.6 after that clean fingered touch the film lightly at varying intervals of time. The films are considered dry when no appeared marks are left by the fingerprint on the film in the same area on each observation. The dried films thickness was 12.5±2 μm.

Figure 4.5 Humidifier in control room.

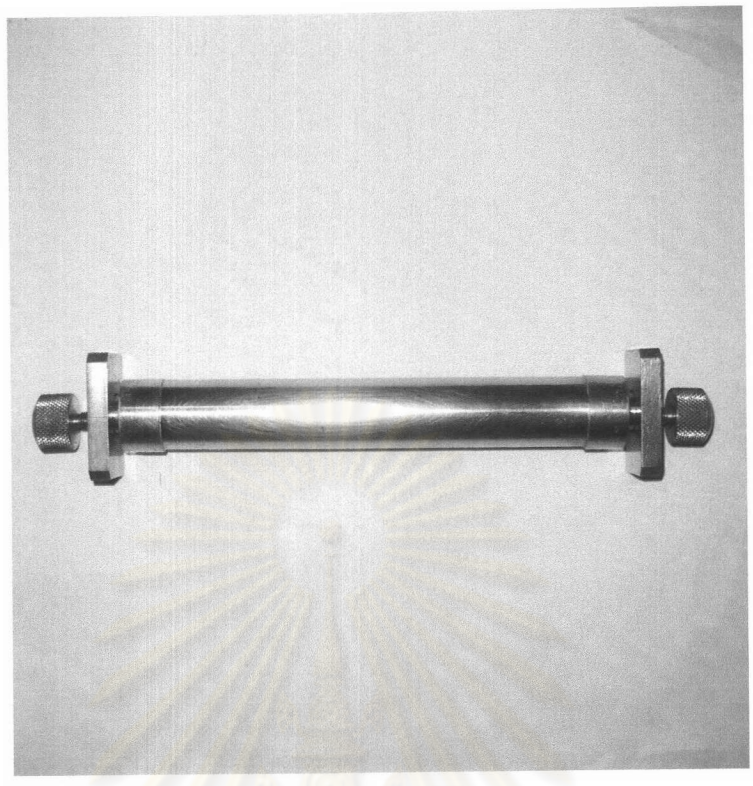


Figure 4.6 Film applicator.

4.1.3 Adhesion

This experiment was conducted following ASTM 3359. The cellulose nitrate solution at amount 18 gram was applied on the glass plate, which cleaned by hydrochloric acid at 5 wt% concentration, let films dried at room temperature for 24 hours. Dry films thickness was up to and including 50 μm space the cuts 1 mm apart and make eleven cuts in horizontal and vertical providing 100 squares of films as illustrate in Figure 4.7. All cuts were about 20 millimeter long by cutting through the film to the substrate in one steady motion using just sufficient pressure on the razor to have the cutting edge reach the substrate, used razor have to use one time for one sample. After making the required cuts brush the film lightly with a soft brush or tissue to remove any detached flakes or ribbons of coatings. Examine the cutting edge and, if necessary, remove any flat spots or wire-edge by abrading lightly on a fine oil stone. Make the additional number of cuts at 90° to and centered on the original cuts. Brush the area as before and inspect the incisions for reflection of light from the glassplate. Remove two complete laps of tape and discard. Remove an additional length at a steady rate, no jerking, and cut a piece about 75 millimeter long. Place the

center of the tape over the grid and in the area of the grid smooth into place by a finger. To ensure good contact with the film rub the tape firmly with the eraser on the end of a pencil. The color under the tape is a useful indication of when good contact has been made. Within 90 $\pm$ 30 s of application, remove the tape by seizing the free end and rapidly, not jerked, back upon itself at as close to an angle of 180° as possible. Inspect the grid area for removal of coating from the substrate or from a previous coating using the illuminated magnifier. The percent adhesion was the total adhered squares on the glass plate.

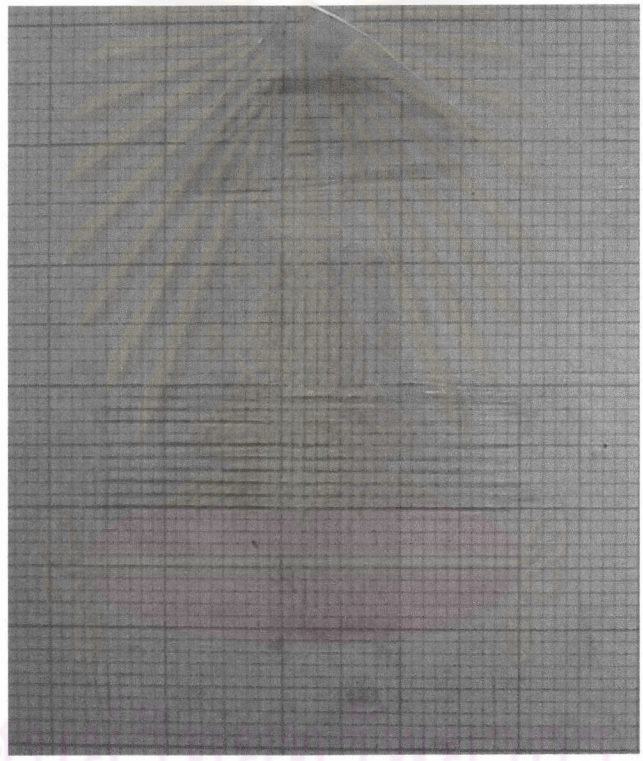


Figure 4.7 Sample of adhesion.

4.1.4 Gloss

This experiment, following ASTM 523, measured the specular gloss of cellulose nitrate films using glossmeter geometries of 60, 20, and 85° as shown in Figure 4.8 (Gloss meter model: Microsheen 250). The 20 gram of cellulose nitrate solution was applied at dimension 75 by 150 millimeter on the black glass plate and allowed film dried at room temperature, after 24 hours, measuring gloss of film compared with the gloss of standard black glass. Put the geometry sensors of 60, 20,

and 85 ° on the dried films, the gloss value of them were shown on the monitor screen.

Figure 4.8 Gloss meter.

4.1.5 Hardness

The cellulose nitrate solutions in amount of 3-5 gram were applied on the slide glass and dried at room temperature for 24 hours, after that they were measured hardness by microhardness tester as depicted in Figure 4.9. The 100 gram force of test load was applied on the films surface for 15 seconds and measured diagonal length by optical microscope and recorded dimension for calculating hardness value in unit of vicker.

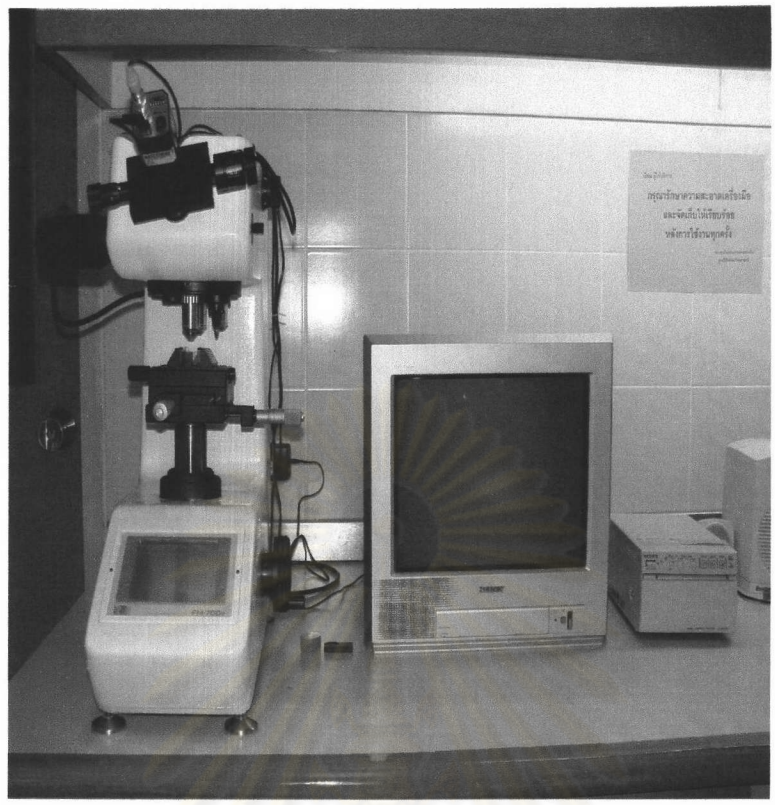


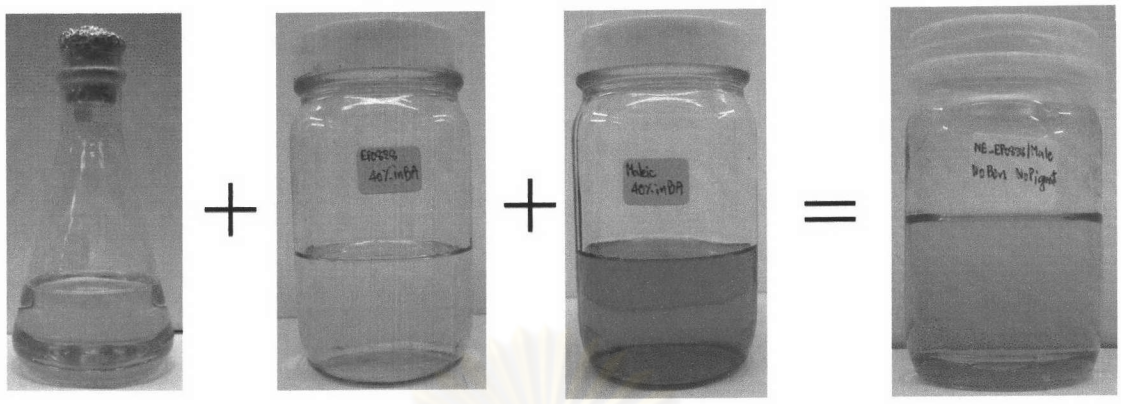
Figure 4.9 Microhardness tester.

4.1.6 Surface texture

The surface texture of dried cellulose nitrate films were studied with ISM-5400 scanning electron microscope at an acceleration voltage of 10 kV. The dried films were coated with thin film of gold using JEOL model JFC-1100E Ion Sputtering Device before obtaining the micrograph.

4.2. Clear nail enamel

Nail enamel solution was obtained by mixing 10 grams of resin, 5 grams of a DBP plasticizer, and 50 grams of a cellulose nitrate solution (contained cellulose nitrate of 10 grams, ethyl alcohol of 5 grams, butyl acetate of 15 grams, and 20 grams of ethyl acetate). The secondary film formers used are acrylic SD603 (Siam Chemicals. Co.,Ltd), silicone, benzoxazine, maleic, and epoxy. A plasticizer is dibutyl phthalate (MC Industrial Chemical Co.,Ltd.). All of clear nail enamel films were tested adhesion by cross cut showing the suitable resin type for nail enamel. Effect of various epoxy and maleic content also was investigated as illustrated in Figure 4.10.



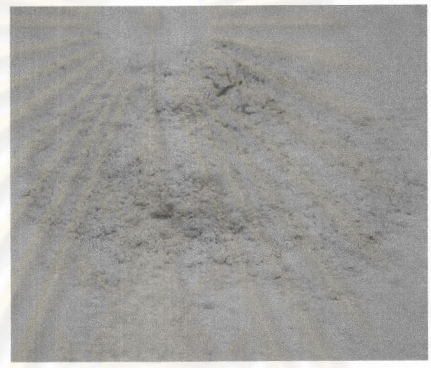
CN solution | Epoxy 40% in butyl acetate | Maleic 40% in butyl acetate | Clear nail enamel

Figure 4.10 Clear enamel preparation.

4.3. Thixotropic agent preparation and its solution characterization

In Figure 4.11a, the dimethyl dioctadecyl ammonium montmorillonite clay (Bentone38 from Rheox, Inc.) was heated up at 100°C for 1 hour to remove moisture and stored in a descicator. The clay was dispersed at the amount of 8 gram in 100 gram of toluene using a mechanical stirrer, as shown in Figure 4.12, at room temperature for 4, 8, 14, 24 and 48 hours with a constant speed 650 rpm. Another dispersion preparation is by using a homogenizer, as shown in Figure 4.13, which was conducted at room temperature at high shear rates of 6500, 9500, 13500, 21500, and 24000 rpm for 5 minutes at each speed. The thixotropic behavior was characterized by a rotational viscometer (Physica Rheolab MC1: Standard measuring system MS-Z3 DIN/MC1) to compared homogenizing suspension with motor stirring suspension. The other two different organoclays such as dimethyl benzyl dodecyl ammonium hectorite (bentone27: Figure 4.11b) and dimethyl dioctadecyl ammonium bentonite (bentone34: Figure 4.11c) were also prepared suspension gel for nail enamel as shown in Figure 4.14.

(a)



(b)

(c)

Figure 4.11 Organoclays powder:
(a) bentone38, (b) bentone27, and
(c) bentone34

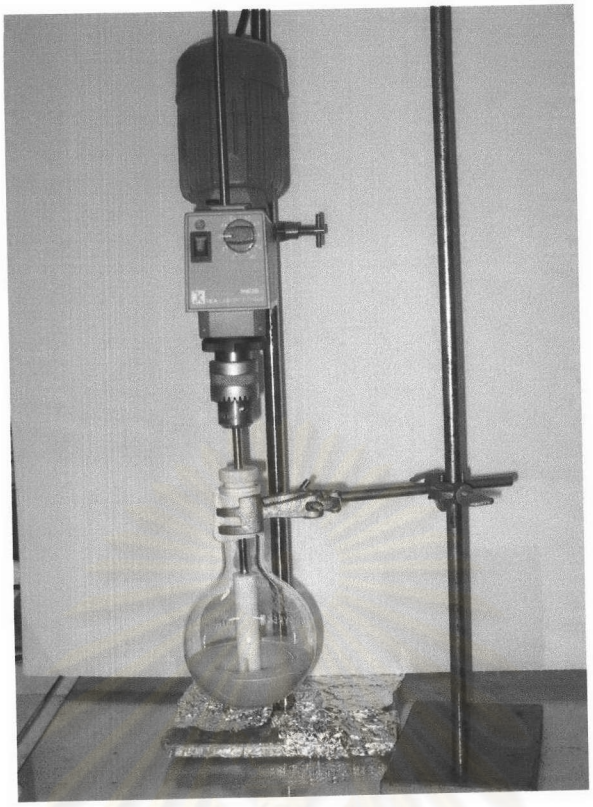


Figure 4.12 Mechanical stirring.

Figure 4.13 Homogenizing.

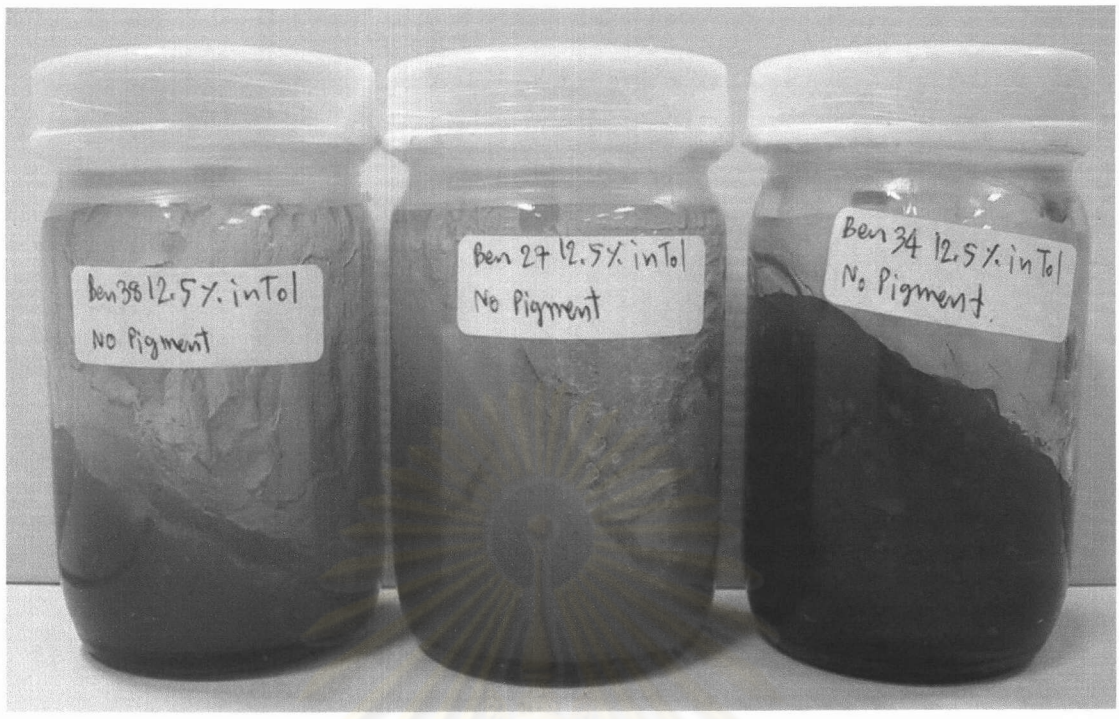


Figure 4.14 Suspension gels of three organoclay.

4.4 Preparation of polymer/clay nanocomposite casted Sheet

The polymer/clay nanocomposite was prepared by mixing clear nail enamel as received from 4.2 with organoclays suspension as received from 4.3 adjusted the organoclay content in final formula to be 0.35, 1, 3, and 5 wt% by loading in container and mixed for 5 minutes using homogenizer at 9500 rpm. Interlayer spacing of organoclay in nanocomposite was investigated by XRD as shown in Figure 4.15. Sample was detected by X-ray diffractometer of Bruker model D8 ADVANCE with CuK α radiation (1.541 Å). The voltage and the current are 40 kV and 30 mA, respectively. The measurement was scanned at 2θ in range of 1.0 to 15.0 with scan speed 30.0 sec/step and step size 0.05 ^{o}2θ. The measurements were operated by EVA program.

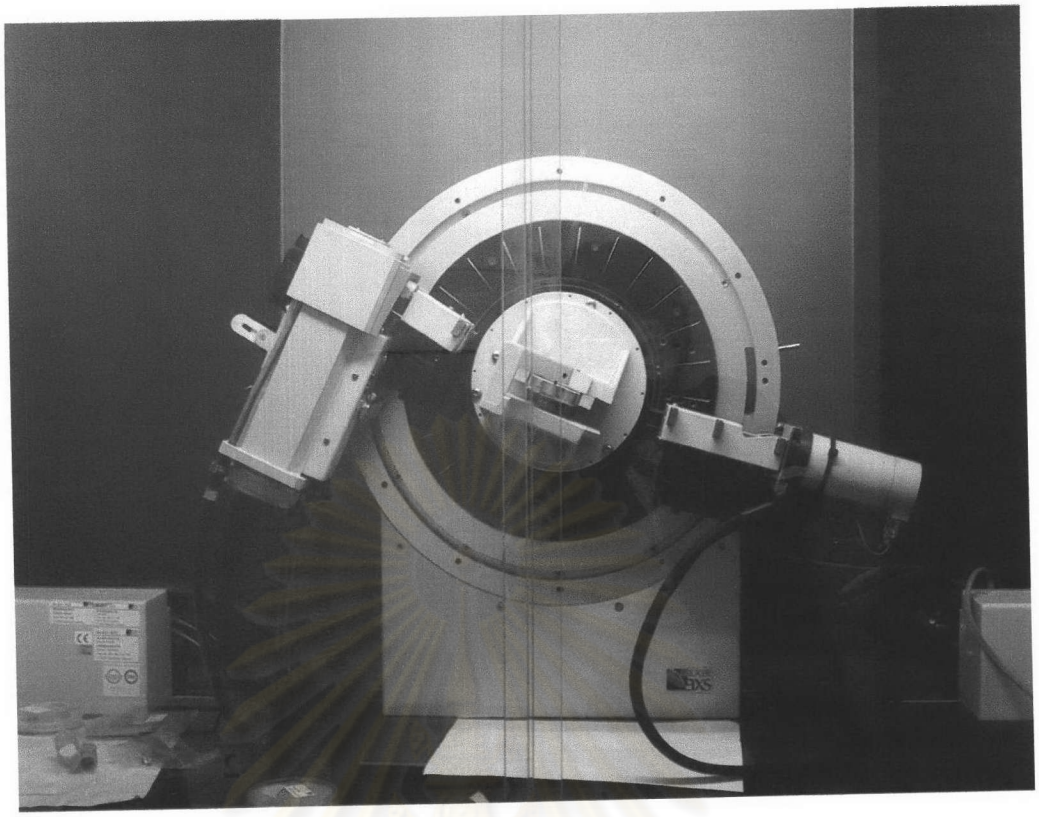


Figure 4.15 X-ray diffraction.

4.5. Color nail enamel

In this process, the amount of toluene of 34 grams was used to suspend the bentone38, bentone27, and bentone34, which prepared from 4.2, were mixed with 65 grams of the clear nail enamel solution. The pigment suspended system was conducted by adding the desired amount of mica coated titanium dioxide pigment (Novant Chemicals Co.,Ltd) at 1 grams in the nail enamel using a mechanical stirrer (at 600 rpm for 1 hour) and a homogenizer (at 9500 rpm for 5 minutes). The solid films were obtained by casting the enamel solution on a clean glass plate followed by evaporating the solvent in an ambient atmosphere to yield films of constant weight. The scanning electron microscope was used to compare surface texture of two different methods. The finished nail enamels of three type organoclay as illustrated in Figure 4.16 were examined the characteristics of the solution including flow behavior, drying time, and film properties such as gloss. The anti-settling system was also investigated by sedimentation. The various organoclays content of 1, 3, 4, and 5wt% were contained in cylindrical flask of 8 milliliter without shaking for 10 days observed the pigment precipitation. The nail enamels which have superior anti-

settling content of organoclay were tested on 10 women. After those of women applied formulated nail enamels, the satisfactions of women on nail enamel characteristics such as pick up, ease of brushing, drying time, and gloss were investigated in questionnaires. The women were allowed to work in everyday life and prohibited to remove nail enamel films before accomplish the research. After 10 days, the abrasion characteristic was examined again in questionnaires. The data of questionnaires was calculated to be percentage of human satisfaction.

Figure 4.16 Color nail enamel.