CHAPTER III

EXPERIMENTAL

3.1 Materials

Flame retardant filler such as APP was purchased from Clarient Co.,Ltd. ATH was attained from Nakaze Chemie. The raw material: 100% acrylic emulsion latex was purchased from Dow Corning, anti-foaming surfactants were purchased from BYK-Chemie.

Antifungal additives: the existing one is zinc omadine from Arh Chemical. The tested antifungal agents such as berberine chloride from Fluka; Captan and iprodione were from Service Chem.

3.2 Flammability and anti-fungal measurement

The OI value was determined according to ISO-4589 Part 2 at Thailand Institute of Scientific and Technological Research (TISTR).

The specimens were conditioned at 23°C and 50%RH for at least four days prior to testing by Fire Testing Technology (FTT) Oxygen Index Apparatus. OI method which describes the tendency of a material to sustain a flame, was widely used as a tool to investigate the flammability of polymers. They provide a convenient, reproducible, means of determining a numerical measure of flammability. These methods have been used to systematically investigate the relative flammability of fire-retarded materials, frequently comparing the effectiveness of fire-retardants and fire-retardant mechanism. The quintessential feature of oxygen index methods is that the sample is burnt within a controlled atmosphere. The standard procedure is to ignite the top of the sample, using a gas flame which is withdrawn once ignition has occurred, and to find the lowest oxygen concentration in an upward flowing mixture of nitrogen and oxygen which just supports sustained burning. The criticality typically takes the form of a minimum burning length; either specifying that the sample must burn for a certain length of time or that a specified length of material be consumed. The effectiveness of fire retardants is measured by the change in the critical oxygen concentration that they induced as a function of their concentration. Polymers with OI higher than 26 are considered to be self-extinguishing in general [3].

Antifungal activity was tested following Thai Industrial Standard 285 Volume 21,1982, at Thailand Institute of Scientific and Technological Research (TISTR).

The specimens were kept in sterilized room for 48 hours before testing the fungal growth by incubating at 28-30°C, %RH 85-90 for minimum 7 days. Test specimens which no growth of fungi in the focus area are considered to be resistant to fungus.

3.3 Fineness of wet paints (Follow TISI 285 Vol. 8)

Use the fineness guage (Fig. 3.1) to specify the fineness of wet paints.

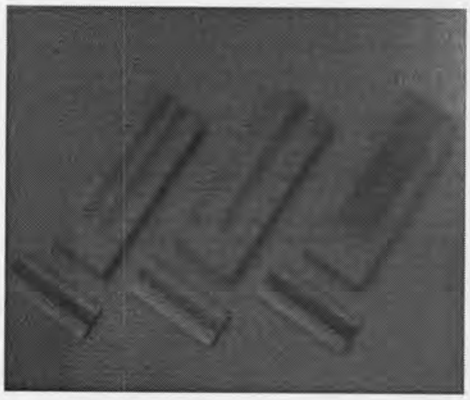


Figure 3.1 Fineness guage.

3.4 Viscosity of flame retardant paints

Content of inorganic flame retardant fillers may affect on viscosity of finished paints. With too low viscosity, sagging problems were observed while with too high viscosity the difficulty in application was faced. Rotor thinner viscometer (Fig. 3.2) was used for determination the viscosity of paint.

To control the viscosity into standard range, thickener should be add for lower viscosity and water adjustable for higher viscosity.

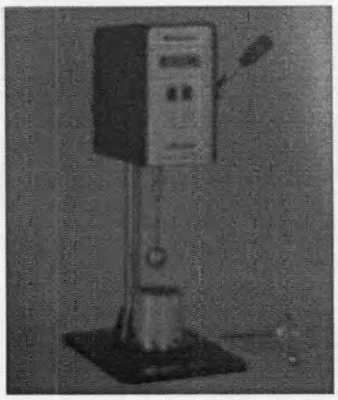


Figure 3.2 Rotor Thinner.

3.5 Opacity of flame retardant paints (Follow TISI 285)

The presence of titanium dioxide (TR-92) in formulation showed good hiding power (good opacity) when it was covered on the substrate. APP and ATH were good flame retardant inorganic fillers but poor opacity property. The good decorative paints should have good opacity to conceal the substrate. This research tries to optimize the formula to get both properties.

The opacity meter (Fig 3.3) was used to specify the opacity data of paints in each formulation. Wet paint was applied on black-white paper by control thickness application (Thickness 100 µm). Leave it dry at room temperature for 30 min. Then opacity meter was used to specify the opacity data of paints in each formulation.

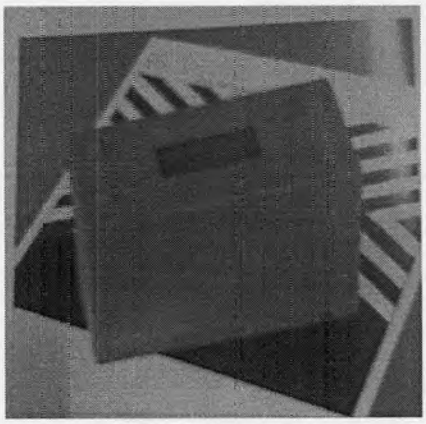


Figure 3.3 Opacity Meter.

3.6 Bending resistance test (Follow TISI 285 Vol.19)

Wet paints were coated onto the tin plate by control thickness applicator and left it for 7 days before bend test by mandrel (Fig. 3.5) with diameter 6 mm for interior paint.

Figure 3.4 Mandrel bend tester.

3.7 Water resistance (Follow TISI 285)

Wet paint was applied onto the fiber cement panel 2 coats by brushing and left it for 7 days before aging into the water for 18 hrs.

3.8 Washability (Follow TISI 285)

Wet paints were coated onto the mirror (thickness 150μ) and leave it for 4 days after that aging at 60^{o}C for 1 day. Apply standard carbon black onto the dry paint film and leave for 30 min. Then bake at 105^{o}C for 30 min before wash ability testing by wet scrub machine 100 cycles. The web scrub machine is presented as shown in Fig. 3.4.

Figure 3.5 Wet scrub machine.

3.9 Apparatus

- High speed mixing machine for paint mixing.
- Fire Testing Technology (FTT) Oxygen Index Apparatus for flammability testing.
- Film applicator by Sheen instruments Ltd. was shown in fig.A1
- Wet abrasion scrub tester model 903 by Sheen instruments Ltd.
- Digital Rotothinner model 455N by Sheen instruments Ltd.
- pH meter
- Opacity meter model 310 by Sheen instruments Ltd.
- Fineness of grind gauges model 502 single channel version by Sheen instruments Ltd. for checking the particle size.

3.10 Paints preparation procedure

In this study, 2 types of flame retardant fillers: ATH and APP were used to mix into the paints by high speed mixing machine before investigating the flame retardant property.

First step, mix water, propylene glycol (PG) and additives together by slow speed for 1 min. Then slowly add the filler including flame retardant fillers following formulations **1-12** and stir well for 2-3 min. After that thickener was added and followed by some water and ammonia solution then slightly increase the mixing speed and check the particle size after 10 min. After getting the particle size, reduce the mixing speed and add the rest ingredients following the formulation in Table 3.1.

Table 3.1 The flame retardant paint formulations.

Descriptions	Function	Flame retardant development formulations						
		Existing	**2**	**3**	**4**	**5**	**6**	**7**
Water	Solvent	55.0	55.0	55.0	55.0	55.0	55.0	55.0
PG		5.5	5.5	5.5	5.5	5.5	5.6	5.5
Orotan 681	Dispersing	4.0	4.0	4.0	4.0	4.0	4.1	4.2
Tergitol NP9	Surfactant	1.1	1.1	1.1	1.1	1.1	1.2	1.0
Foamaster	Defoamer	1.0	1.1	1.1	1.2	1.0	1.0	1.1
Tioxide TR92	Filler	108.5	78.5	28.65	28.5	50.0	18.5	50.0
Kaolin AK35	Extender	20.0	-	-	-	-	-	-
APP	FR	-	50.0	100.0	50.0	-	50.1	150.0
ATH	FR	-	-	-	50.0	78.5	50.0	-
Pentaerytritol	Carbon source	-	-	-	-	-	10.0	-
Tylose	Thickener	1.0	1.0	1.0	1.0	1.0	1.0	1.0
Water	Solvent	5.13	5.5	5.6	5.0	5.0	5.1	5.2
AMP 95		1.3	1.4	1.3	1.6	1.0	1.3	1.4
Water	Solvent	60.6	61.5	60.8	61.2	58.5	60.2	59.3
Foamaster NDW		2.0	2.0	2.0	2.3	2.7	2.3	2.1
Texanol	Coalescing	6.6	6.5	6.7	6.7	6.6	6.8	7.1
Acticide HF	Biocides	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Zinc Omadine	Fungicides	5.0	5.0	5.0	5.0	5.0	5.0	5.1
Foamaster SA3	Defoamer	2.0	2.0	2.0	2.0	2.0	2.0	2.0
Ucar Latex	Binder	175.0	175.0	175.5	175.0	175.9	2.0	2.0
Ropaque Ultra		29.7	29.8	29.8	30.3	30.2	175.0	103.8
Acrysol		5.1	5.2	5.3	5.3	5.3	31.8	29.8
PG		8.8	8.2	8.4	8.5	8.5	5.1	5.4
Rheolate 430		2.5	2.6	2.6	3.4	3.4	8.9	8.1
Water		3.7	3.2	3.3	2.5	2.5	2.7	2.8
AMP 95		-	-	-	-	-	-	-
Total		504.0	504.6	505.2	501.4	505.6	503.2	502.4

Table 3.1 (Continued)

Descriptions	Function	Flame retardant development formulations				
		8	**9**	**10**	**11**	**12**
Water	Solvent	55.0	55.0	55.0	55.0	55.0
PG		5.5	5.5	5.5	5.5	5.5
Orotan 681	Dispersing	4.0	4.0	4.1	4.0	4.1
Tergitol NP9	Surfactant	1.3	1.1	1.1	1.1	1.1
Foamaster	Defoamer	1.1	1.0	1.0	1.0	1.0
Tioxide TR92	Filler	50.0	50.0	50.0	50.0	50.0
Kaolin AK35	Extender	-	-	-	-	-
APP	FR	125.0	100.0	50.2	50.0	50.2
ATH	FR	25.0	50.0	75.0	50.0	75.0
Pentaerytritol	Carbon source	-	-	-	-	-
Tylose	Thickener	1.0	1.0	1.0	1.1	1.0
Water	Solvent	5.5	5.1	5.0	5.0	5.0
AMP 95		1.5	1.2	1.4	1.5	1.4
Water	Solvent	60.3	59.8	59.3	59.3	59.3
Foamaster NDW		2.0	2.0	2.0	2.1	2.0
Texanol	Coalescing	6.6	7.0	6.6	6.6	6.6
Acticide HF	Biocides	0.6	0.7	0.5	0.7	0.5
Zinc Omadine	Fungicides	5.2	5.0	5.0	5.3	-
Captan	Fungicides	-	-	-	-	5.0
Foamaster SA3	Defoamer	2.0	2.0	2.0	2.1	2.0
Ucar Latex	Binder	103.7	113.8	133.7	155.4	133.7
Ropaque Ultra		30.6	19.8	25.1	30.0	25.1
Acrysol		5.8	5.0	5.2	5.2	5.2
PG		8.3	8.6	8.5	9.0	8.5
Rheolate 430		2.5	2.5	2.7	3.8	2.7
Water		1.9	2.5	2.5	-	2.5
AMP 95		-	-	-	-	-
Total		505.6	504.4	502.6	506.9	503.7

In this research, flame retardant property was improved by varying the content of inorganic flame retardant filler: APP and ATH in 100% acrylic emulsion paint.

Existing product contains TiO_2 and kaolin as inorganic fillers. The total filler content is 26% consisting of TiO_2 22% and kaolin 4%

Formulations 2,3 were the flame retardant acrylic paints improvement by adding APP 10% and 20%, respectively at the total filler content at 26%.

Formulation 4 was prepared to observe the synergistic effect between APP and ATH. The mixing ratio is 1:1 (10% : 10%).

Formulation 5 contained ATH 16%, test compare with existing product. From this formulation we can observe effect of ATH on OI value.

Formulation 6 was compared with formulation **4** to study the effect of pentaerytritol without spumific agent that used for intumescence paint.

Formulations 7, 8 and 9 containing 10% TiO_2 but different ratios of APP:ATH were prepared to observe synergist of APP and ATH.

Formulations 10, 11 were prepared to improve the water resistant property by reducing flame retardant filler content from 30% (in formulations **7**, **8**, **9**) to 20 and 25%

Formulation 12 was similar to formulation **10**, except for replaceing zinc omadine antifungal with captan.

3.11 Test specimens preparation and procedure for oxygen index testing

For paint specimens was prepared in the forms of flexible film or sheet (Test specimen form V in Table 3.2).

Table 3.2 The test specimens dimensions for any application.

Test Specimen Form	Dimension(mm.)			Typical Use
	Length	Width	Thickness	
I	80-150	10 ± 0.5	4 ± 0.25	For moulding material
II	80-150	10 ± 0.5	10 ± 0.5	For cellular materials
III	80-150	10 ± 0.5	10.5 max.	For sheet materials as received
IV	70-150	6.5 ± o.5	3 ± 0.25	self-supporting moulding sheet materials for electronic purposes
V	140	52 ± 0.5	10.5 max.	For flexible film or sheet

The testing machine for oxygen index (OI) value following ISO-4589 Part 2 was depicted in Fig. 3.6.

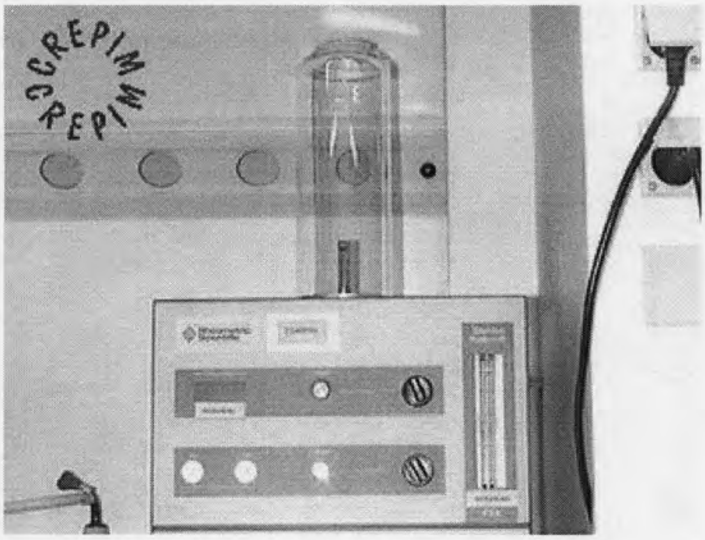


Figure 3.6 ISO 4589-2 Oxygen Index appliance.

3.12 Antifungal paint formulation

To evaluate new antifungal agents blending in latex paints (100% acrylic latex), the mixing step followed the same procedure as that for flame retardant testing except the new antifungals such as propyl 4-hydroxybenzoate, berberine chloride, captan, iprodione and 2,4-dichlorocinamic acid were replaced the existing one (zinc pyrithione).

Anti-**2** and **3** were prepared to study the effect of propyl 4-hydroxybenzoate in the paint formulation by vary in 0.5 and 1.0% content.

Anti-**4, 5, 6 and 7** were evaluated the antifungal property by different antifungal agents such as berberine chloride, captan, iprodione and 2,4-dichlorocinnamic acid, respectively.

Formulation **12** was replaced the existing antifungal by captan at 1% content by weight. This formulation was manipulated to compare with anti-**5** which used captan at 0.5% by weight.

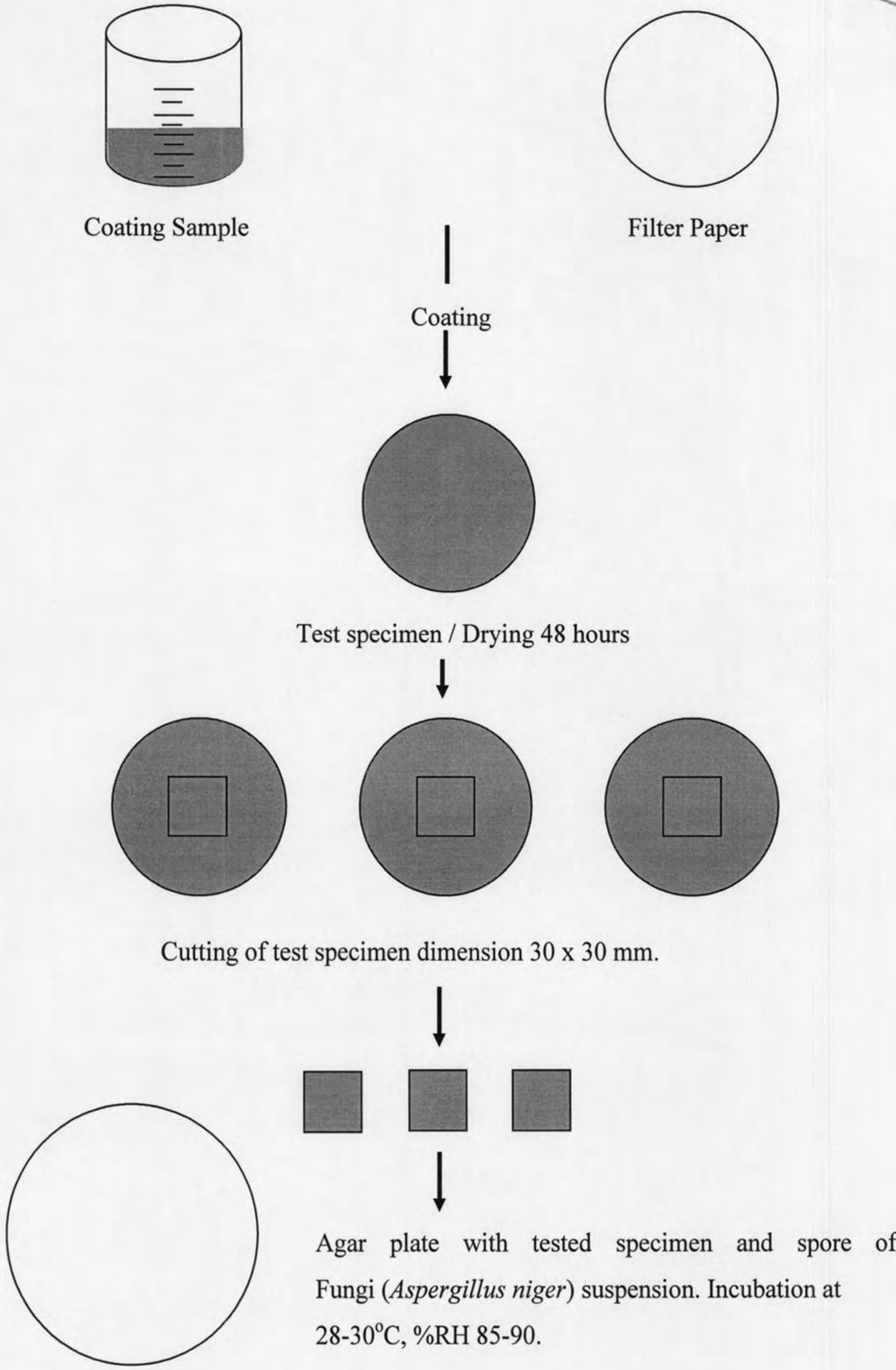
Table 3.3 Paint formulation for new anti-fungal

Descriptions	Sample No. / Function	New Anti-fungal paint formulation: Existing	Anti-2	Anti-3	Anti-4	Anti-5	Anti-6	Anti-7	F.12
WATER	Solvent	11.0	11.0	11.0	11.0	11.0	11.2	11.1	11.1
PROPYLENE GLYCOL		1.1	1.2	1.1	1.2	1.1	1.1	1.2	1.2
OROTAN 681	Dispersing	0.8	0.8	0.8	0.9	0.8	0.8	0.8	0.8
TERGITOL NP -9	Surfactant	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
FOAMASTER SA -3	Defoamer	0.2	0.2	0.2	0.2	0.3	0.2	0.3	0.3
TIOXIDE TR 92		21.7	21.5	21.7	21.7	21.8	21.9	21.7	21.9
KAOLIN AK 35		4.0	4.0	4.0	4.1	4.0	4.0	4.0	4.0
TYLOSE H 15000 YP2	Thickener	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
WATER	Solvent	1.5	1.5	1.5	1.6	1.8	1.7	1.9	1.8
AMP 95		0.3	0.3	0.3	0.3	0.3	0.2	0.3	0.3
WATER	Solvent	11.9	11.9	11.9	12.3	11.8	12.1	12.1	12.3
FOAMASTER NDW		0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4
TEXANOL /IBT	Coalescing	1.3	1.3	1.3	1.4	1.3	1.5	1.4	1.4
ACTICIDE HF	Biocides	0.1	0.1	0.1	0.2	0.1	0.1	0.1	0.1
ZINC OMADINE	Fungicides	0.5	-	-	-	-	-	-	-
Propyl 4 Hyroxy Benzoate	Fungicides	-	0.5	1.0	-	-	-	-	-
Berbirine Chloride	Fungicides	-	-	-	0.5	-	-	-	-
Captan	Fungicides	-	-	-	-	0.5	-	-	1.0
Iprodione	Fungicides	-	-	-	-	-	0.5	-	-
2,4 Dichlorocinamic acid	Fungicides	-	-	-	-	-	-	0.5	-
FOAMASTER SA -3	Defoamer	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4
UCAR LATEX R 361 G	Binder	34.9	35.0	34.9	35.0	35.3	35.2	35.6	35.6
ROPAQUE ULTRA		6.0	6.0	6.0	6.1	6.4	6.3	6.2	6.3
ACRYSOL RM -2020 NPR		1.0	1.1	1.0	1.1	1.0	1.0	1.1	1.1
PROPYLENE GLYCOL		1.6	1.6	1.6	1.6	1.7	1.7	1.8	1.8
RHEOLATE 430		0.5	0.5	0.5	0.6	0.6	0.5	0.6	0.6
WATER		0.5	0.5	0.5	0.1	0.7	0.1	0.5	0.5
AMP 95		-	-	-	-	-	-	-	-
Total		100.0	100.1	100.5	100.9	101.7	101.5	102.2	103.0

*These paint formulations are 100% acrylic emulsion for interior. The existing anti-fungal is zinc omadine.

3.13 Anti-fungal testing procedure

The testing procedure refers to that reported in Thai Industial Standard 285 Volume 21, 1982. [15] The general scheme is presented in Scheme 3.1.



Scheme 3.1 The determining resistant to fungal growth.