

## CHAPTER FIVE

### GENERAL DISCUSSION AND CONCLUSION

#### 5.1 PURIFICATION OF CNSL:

The study reveals that the method adopted in this work to purify the raw CNSL 1) using fullers earth as a purifier and 2) using animal charcoal as a purifier gave promising results. (see tables 2.2 & 2.3 ) The purified CNSL show better clarity than the raw CNSL, though the dark brown color remains unchanged. However the purified CNSL has been found to be unsuitable for application in surface coating, because of it's non-drying nature, even with the driers.



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#### 5.2 POLYMERIZATION OF (P.D) CNSL WITH 40% FORMALDEHYDE:

The following factors have been carefully controlled during the experimental method of polymerization of CNSL with Formaldehyde.

##### I. Acid value of (P.D) CNSL

It was found that P.D CNSL with acid value less than 5mg of KOH /1gm of P.D CNSL caused gellation in the reacting system. It is also known fact that the acid value of P.D CNSL grater than 15 mg. Of KOH/ 1 gm of P.D CNSL is not suitable

for manufacturing resin in paint industry. Therefore acid value of P.D CNSL is preferred in the region of 5\_15mg of KOH/ 1 gm of P.D CNSL for manufacturing resin in paint industry. As a result acid value of purified CNSL was chosen less than 15mg of KOH/1gm of P.D CNSL for polymerization.

## II. Viscosity Control :-

A rapid increase in viscosity was observed with the rising of temperature & time. To prevent this increase in viscosity, the reaction was carried out at low temperature in the range 85 ~ 100<sup>0</sup>c, under N<sub>2</sub> atmosphere

## III. Water content :-



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It was observed that the water content even in the presence of small % had detrimental effect on the quality of the polymerized resinous product. As a result water content in the polymerized system was reduced to minimum quantity as possible by applying suction with a vacuum unit.

### **5.3 USE OF POLYMERIZED CNSL /HCHO BASED RESIN IN COATING INDUSTRY:**

#### **a) IN AIR DRYING VARNISH:-**

It has been found that the quality of polymerized (P.D) CNSL based Varnish with lead and cobalt driers is comparable to that of 70% Soya alkyd resin based varnish in terms of drying time, Adhesion, Gloss, Gloss retention & scratch resistance. (see tables 4.8(a)&4.8(b)) The colour obtained of polymerized (P.D) CNSL varnish is yellowish brown & is more attractive than the varnish coating prepared with 70% Soya alkyd resin. Hence the varnish prepared with polymerized CNSL resin can be used as an air drying varnish with 12% cobalt and 36% Lead driers in local industries.

#### **b). IN STOVING VARNISH.**

From the result of table 4.9 it has been found that the quality of polymerized resinous product of CNSL based varnish is almost similar to that of melamine resin based varnish in terms of scratch resistance adhesion, and resistance to alcohol but the gloss of the coating has been observed to be some what lower than that of melamine resin based varnish. This could be attributed to the excess free formalin presence in the polymerized resinous product of CNSL.

## 5.4 SUGGESTIONS FOR FUTURE WORK:

### 1) DECOLORISATION OF CNSL BY CHLORINATION:

In the purification of CNSL the purified CNSL has been found to be not clear. As this color may affect the color of the end products, some method has to be adopted to improve the color. It is suggested that bleaching with chlorine gas would be valuable to bring about this change.

### 2) PURIFICATION OF RAW CNSL

In the purification process it can be tried with cheaply available china clay instead of fuller earth or animal charcoal, this would become more economical process, in the long run.

### 3) POLYMERIZATION OF CNSL WITH FORMALIN

In polymerization Para formaldehyde could be used instead of formaline because of its concentrated form. This would help to reduce the water content, which in turn would give desirable results in drying time of coating and gloss of the coating.

### 4) DECARBOXYLATION (REFINING)

As the thermal polymerization occurs simultaneously with decarboxylation process at high temperatures it is suggested that the temperature of decarboxylation should be maintained at low level preferably below  $140^{\circ}\text{C}$ , though it takes longer time for the decarboxylation.

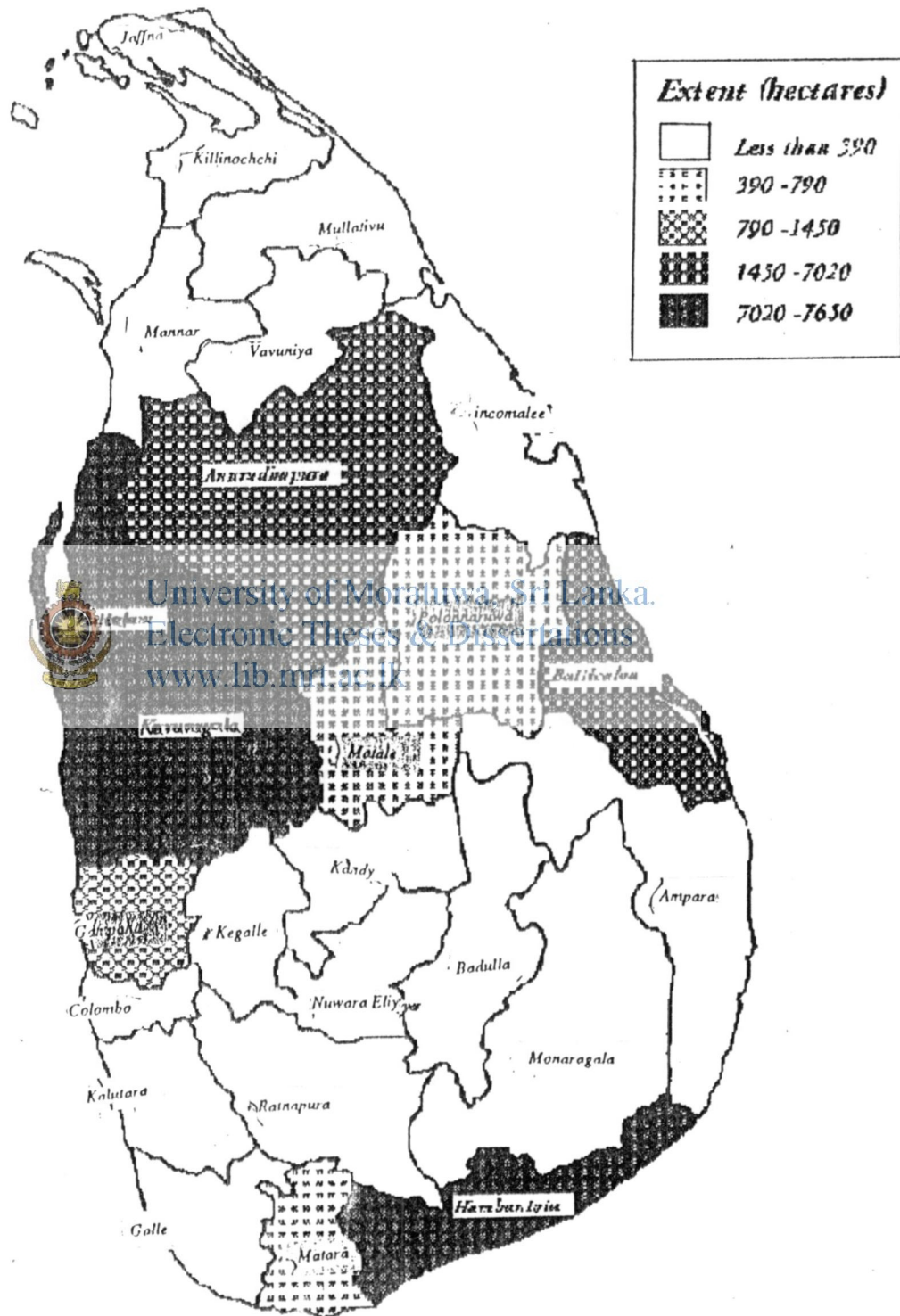


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## Extent under cashew cultivation by district: 1996



Source: Department of Census and Statistics



## APPENDIX (B)

### 1.0 DETERMINATION OF SPECIFIC GRAVITY OF CNSL:

Pyknometer (specific gravity bottle) is cleaned, dried and filled with freshly boiled and cooled distilled water. Bottle is filled to overflowing by taking care to prevent entrapment of air bubbles. The stopper is inserted and bottle is immersed in water bath. The entire bulb is completely covered with water and held at constant temperature of 30<sup>0</sup> C for 30 minutes. Bottle is removed from the bath wiped from out side and dried. The bottle is weighed with it's content.

Bottle is cleaned and dried. It is then completely filled with the sample of CNSL. The stopper is inserted and the bottle is weighed with it's contents. The specific gravity is determined using following expression.



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$$\text{Specific gravity} = \frac{(W_2 - W_0)}{(W_1 - W_0)}$$

Where

$W_0$  = Weight of Empty Pyknometer

$W_1$  = Weight of Pyknometer + Water

$W_2$  = Weight of Pyknometer + Sample

### 1.1 DETERMINATION OF ACID VALUE OF CNSL <sup>23</sup>

About 1 gm of the sample is weighed accurately into a 250ml capacity conical flask. It is dissolved in 50ml of neutral Xylene / Methanol mixture (xylene /



methanol mixture is prepared by mixing equal volumes of xylene & methanol) 0.5 ml of bromothymol blue indicator solution is added to the mixture is titrated with N alcoholic potassium hydroxide till the solution colour change from brownish yellow to greenish blue.

The acid value is calculated as:

$$\text{Acid value} = \frac{V * N * 56.1}{W}$$

expressed in mg of KOH / gm of sample

Where      V = ml of standard alcoholic KOH required  
               N = Normality of Alcoholic KOH  
               W = Weight of the solid sample

## 1.2 DETERMINATION OF IODINE VALUE OF CNSL (BY WIJ'S METHOD):

### PREPARATION OF STOCK SOLUTION <sup>23</sup>

Stock solution is prepared by dissolving 13.4 or 0.1 gm of sublimed Iodine to one litre of glacial acetic acid using gentle heat. Then 50 to 100 ml of the solution is kept a side & chlorine gas which is generated, by heating manganese dioxide with concentrated hydrochloric acid is introduced into the remainder until the characteristic colour change occurred & the halogen content is maintained nearly about double its original value and the halogen content is maintained by adding the requisite quantity of the Iodine – acetic acid solution.

About 0.10 to 0.12 gm of the sample is weighed accurately into 250 ml capacity Erlenmeyer flask & 25 ml of carbon tetra chloride is added. The sample is dissolved in carbon tetra chloride by gentle swirling and if necessary, the content warmed on water bath to dissolve the content and then cooled 25 ml of wijs solution is pipette out into the flask and the flask is then stoppered. Contents are swirled gently and 10 ml of 2.5% mercuric acetate solution in glacial acetic is added to the flask & flask is placed in a dark place away from light for one hour. After one hour the flask is swirled for one or two minutes followed by addition of 50 ml of distilled water. Then 20 ml of 10 % potassium Iodide solution is added. The contents are titrated with

standard 0.1 N sodium thiosulphate solution using 0.5 ml freshly prepared starch solution as an indicator, till the blue colour changed to colour less. The blank is carried out under identical condition the Iodine value is calculated as

$$\text{Iodine Value} = \frac{(B - S) * N * 12.69}{W}$$

Expressed in gm of I<sub>2</sub> absorbed / 100 gm of sample

Where

- B = ml of standard sodium thiosulphate solution required by blank
- S = ml of standard sodium thiosulphate solution required by sample
- N = Normality of standard sodium thiosulphate
- W = Weight of the sample taken.

### 1.3 DETERMINATION OF SPECIFIC GRAVITY OF RESIN (USING SPECIFIC GRAVITY CUP)



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The temperature of the coating mixture is measured and is adjusted to 30<sup>o</sup>c. The clean, dry specific gravity cup with lid is weighed and is recorded to 0.1 gm. Accuracy. The cup is filled carefully avoiding air entrapment and bubble formation and lid is pressed firmly in to the position. If no excess is appeared through the orifice in the lid, a further portion of sample is filled to the content in the cup and the operation is repeated until the cup is completely full. The excess material is removed from the lid and is reweighed to 0.1 gm. accuracy. The specific gravity is determined using the following expression.

$$\text{Specific gravity} = (W_2 - W_1) / (W_1 - W_0)$$

Where  $W_0$  = Weight of the empty specific gravity cup  
 $W_1$  = Weight of the specific gravity cup+ water  
 $W_2$  = Weight of the specific gravity cup+ sample

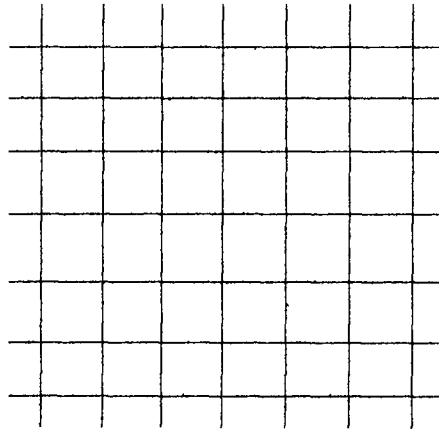
#### 1.4 DETERMINATION OF SCRATCH RESISTANCE OF THE COATING (BY USING WOLFF WILBORN PENCIL):

The instrument consists of a Pencil holder the weight of which is so arranged that the pressure exerted by the pencil point is precisely 300gm. The process is started with the hardest pencil and is continued down the scale of hardness to the pencil that would not scratch the film. This process is continued until a pencil is found that would neither cut through nor scratch the surface of the film. It is observed any defacement of the film other than cut is considered the scratch.

→ Hardness of the pencil is decreased  
 6H, 5H, 4H, 3H, 2H, H, F, HB, B, 2B, 3B, 4B, 5B, 6B

#### 1.5 DETERMINATION OF ADHESION OF COATING:

The coated panel is placed on a firm base and the parallel cuts are made as follows, by means of cross hatch cutter leaving 1 mm spacing between the cuts. Any detached flakes or dust of coating film is brushed with a soft brush after making the required cuts. The center of the tape was placed over the grid and the area of the grid is smoothen into place by a finger. The tape is rubbed firmly with the eraser of the end of pencil, to ensure good contact with the film. The tape is removed by seizing the free end and is rapidly pulled it off at as close to an angle of  $180^\circ$  as possible and then the grid area is inspected for removal of coating from the coated panel. Adhesion is rated in accordance with the scale., (see below picture).



Classification of Adhesion Test Results

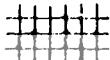
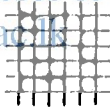

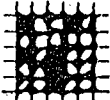
Classification	Surface of cross-cut area from which flaking has occurred. (Example for six parallel cuts)
5B	None
4B	
3B	
2B	
1B	
0B	Greater than 65%

FIG. 1 Classification of Adhesion Test Results



## **1.6 DETERMINATION OF TOUCH DRYING AND HARD DRYING TIME OF THE COATING:**

The test shall be performed at a temperature of  $30 \pm 0.5^{\circ} \text{C}$  and a relative humidity of  $75 \pm 5\%$ .

Note: If these conditions cannot be achieved the temperature and relative humidity at the time of test should be recorded.

A 50  $\mu\text{m}$  bar applicator is placed across width of cleaned glass substrate. The thoroughly stirred sample is placed near one end, across the full width of the glass panel, in front of the applicator. The applicator is drawn down the length of the panel with firm, even pressure at uniform speed. The panel is stood vertically and allowed to dry. The clock is started to measure the drying time as follows.

### **TOUCH DRYING TIME:**

The finger is placed on the coating and is applied very light pressure. If the coating is left without a mark or does not reveal stickiness, the product is said to be touch dry.



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**HARD DRYING TIME:** [www.lib.mrt.ac.lk](http://www.lib.mrt.ac.lk)

The panel is placed in a horizontal position at height such that when the thumb is placed on the coating, and the arm is in a vertical line from the wrist to the shoulder. The film is then drawn down on, using the maximum pressure of the arm, while simultaneously twisting the thumb through  $90^{\circ}$  in the plane of coating. The coating is hard dry if the tearing or any other distortion of the coating.

### **1.7 DETERMINATION OF VISCOSITY (BY FORD CUP NO.4)**

Ford cup is filled with sample maintained at  $30^{\circ}\text{C}$ . The time for the sample to flow out completely is measured in seconds by using stopwatch.

## 1.8 DETERMINATION OF DRY FILM THICKNESS (DFT) OF THE DRIED COATING BY ELCOMETER

The material to be tested is uniformly applied over a mild steel panel and dry. The elcometer is placed on the bare surface of the coated panel and zero value is obtained by pressing zero button. The instrument is then calibrated using to appropriate non magnetic thickness shims which are in the expected range to be measured. The instrument is held firmly on the selected thickness shim that place on the bare part of the steel panel and calibration is carried – out. When that experiment is carried out mechanical vibrations, electric fields and magnetic fields must be avoided. The magnetic tip of the instrument and the surface to be measured should be cleaned and dried. After the calibration is done, elcometer is placed perpendicular to the surface to be tested and readings are taken. Calibration should be checked frequently while the readings are being taken. Sufficient number of readings are taken to characterise the surface.

For laboratory measurements - Three readings for 75\*150 mm  
For field measurements - Five readings for every 10 m<sup>2</sup>



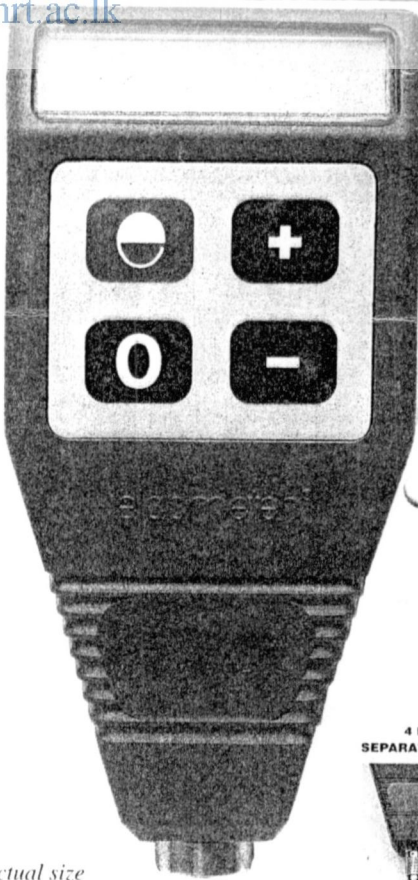
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	<b>ZERO</b>
	<b>CALIBRATE DOWN</b>
	<b>CALIBRATE UP</b>
	<b>LOWEST READING</b>
	<b>HIGHEST READING</b>
	<b>NUMBER OF READINGS</b>
	<b>MEAN</b> Average Thickness
	<b>SIGMA</b> Standard Deviation



Actual size



## 1.9 DETERMINATION OF NON VOLATILE MATTER OF RESIN

About 2+/- 0.5 gm sample is weighed accurately in a petridish and is heated at 130 +/- 5 °c in an oven until all volatile matters is being driven off and sample reach constant weight. The percentage solid content is calculated as follows.

$$\% \text{ Solid content} = (w_1 - w_2) / w_1$$

Where,

$w_1$  = weight of the sample before heating.

$w_2$  = weight of the sample after heating.



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## 2.0. DETERMINATION OF GLOSS OF THE DRIED COATING BY “ TRI- GLOSS METER”

The coating to be tested is applied on the cleaned and dried substrate, to the required thickness. The coated panel is then held in a horizontal position and allow it to dry. After complete drying , the TRI GLOSS METER is placed on the coated panel to measure the gloss.

Note : The geometry plane is appropriated to the degree of gloss level of the applied coating. If the gloss level of the coating is higher( by visual ) then it must be used geometry plane either  $20^{\circ}$  or  $60^{\circ}$ . If the gloss level is very low( almost matt ) then it must be used geometry plane  $85^{\circ}$ .

Once the required geometry is set, the TRI GLOSS METER is placed on the coated surface and the "operate button" is pressed to obtain the gloss value. The reading is expressed as eg. 75%, $60^{\circ}$  where geometry plane is  $60^{\circ}$ .

## Gloss



TRI GLOSS METER

