

Chapter 3 – Methodology

Methodology prevails under the following two sections

1. Determination, organization and preparation of principal requirements

This entailed

- Collection of latex
- Preparation of working plan
- Preparation of a method to supply aeration to latex samples
- Arrange the sample vessels
- Preparation of required chemical solutions
- Preparation of latex samples by varying aeration level and length of maturation.

2. Study of the effect of aeration on property variation of latex with time and air content

This entailed



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- Determination of resistance to destabilization as Mechanical Stability Time
- Determination of acid neutralizing capacity as Alkalinity
- Determination of deterioration in latex in the form of Volatile Fatty Acids Number
- Determination of flow behavior in the form of Viscosity
- Determination of Conductivity

3.1 Determination, Organization and Preparation of Principal Requirements

3.1.1 Selection of Latex

For the purpose of this investigation HA preserved concentrate latex was used. It was carried out two different periods as two different batches. The both batches of latex were collected from the centrifuged plant of the L & C latex (pvt) ltd. Horana.

On the special request made to the centrifuge plant, the latex were obtained without externally added fatty acid soap. After the lattices were obtained from the centrifuged plant of L & C latex Ltd. The properties of the latex were tested before set up the practical. (Table 3)

Aeration was supplied to the latex samples in four different ways. For each different way eight samples were prepared. Four modified samples with air and one control without air gap was prepared as Table 3 and Table 4.

Table 3. Working plan for 1st batch of Latex

Sample code	Aeration level (cm ³ per 1l of latex)	Properties to be investigated					
		MST	Alkalinity	VFA No:	Viscosity	pH	Conductivity
S 1,0	Zero aeration & zero air gap	Yes	Yes	Yes	Yes	Yes	Yes
S 1,1	0 cm ³ /l & air gap*	Yes	Yes	Yes	Yes	Yes	Yes
S 1,2	375 cm ³ /l & air gap*	Yes	Yes	Yes	Yes	Yes	Yes
S 1,3	750 cm ³ /l & air gap*	Yes	Yes	Yes	Yes	Yes	Yes
S 1,4	1125 cm ³ /l & air gap*	Yes	Yes	Yes	Yes	Yes	Yes

* air gap – air: latex ratio= 2:1

Table 4. Working plan for 2nd batch of Latex

Sample code	Aeration level (cm ³ per 1l of latex)	Properties to be investigated					
		MST	Alkalinity	VFA No:	Viscosity	pH	Conductivity
S 2,0	Zero aeration & zero air gap	Yes	Yes	Yes	Yes	Yes	Yes
S 2,1	0 cm ³ /l & air gap*	Yes	Yes	Yes	Yes	Yes	Yes
S 2,2	375 cm ³ /l & air gap*	Yes	Yes	Yes	Yes	Yes	Yes
S 2,3	750 cm ³ /l & air gap*	Yes	Yes	Yes	Yes	Yes	Yes
S 2,4	1125 cm ³ /l & air gap*	Yes	Yes	Yes	Yes	Yes	Yes

* air gap – air: latex ratio= 2:1

The properties investigated were MST, Alkalinity, VFA No, Viscosity, pH and Conductivity.

All the samples were individually tested by four days maturation intervals.



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Table 5:- Properties of the latex before setup the study.

Property	Laboratory test data	
	1 st batch	2 nd batch
Total solid content (%)	62.7	63.34
Dry rubber content (%)	60.69	61.28
Alkalinity	0.68	0.57
MST(sec)	65	70
VFA No:	0.0175	0.0133
Viscosity(cp)	142	148
pH	10.2	10.3
Conductivity (mS/cm)	3.61	3.61

3.1.2 Preparation of other chemicals

All chemicals solutions required for investigate the properties such as Ammonium Hydroxide, Ammonium Sulphate, Barium Hydroxide; Acetic acid, Hydrochloric acid etc. were prepared by using analytical grade reagents accordingly to the methods in ISO standards.

3.1.3 Preparation of latex samples by varying aeration levels and maturation.

Field latex was collected from L & C latex Ltd, preserved and centrifuged in their centrifuge plant in Bellapitiya (Horana). This latex was brought as one batch to the laboratory in ITI laboratory. It was first stained through wire gauze to remove any coagulum particles. They were placed in plastic containers ensuring structure to be same for each group.

Four aeration levels were used as 0 sec, 30 sec, 60 sec and 90 sec, aeration rate $3.75 \text{ cm}^3/\text{sec}$ with a control with zero air gap. Eight samples vessels from one category were prepared and stored under room temperature and one vessel from one aeration level is tested at a time of four days intervals.

Another set of latex was collected from same centrifuged plant and bring them to ITI laboratory and followed the same procedure mentioned above as repeat study.

Sample prepared to investigate property variation with storage time; the date of preparation with the date of investigation carried out; for the properties of the prepared samples, upon maturation are given in Appendix 2



3.2 Methods of Investigation of the Properties of Latex

3.2.1 Determination of MST

MST test of the samples were determined by ISO techniques published by International Standard Organization. Reference No ISO 35:1995(E)

Before determine the MST, TSC and Alkalinity of the latex was carried out in accordance with ISO 124:1992 and ISO 125:1990 respectively

Five latex samples were tested for MST per one day upon aeration level and altogether fourty samples upon maturation.

A test portion of 100 g of latex concentrate from each sample was taken in a glass beaker. That was diluted to $55\% \pm 0.2\%$ TSC with 0.6% NH_3 solution. Without delay, the diluted latex was armed with gentle stirring to $36-38^\circ\text{C}$, using water bath (at 70°C). The diluted sample was immediately filtered through wire gauze and weighed 80.0 ± 0.5 g of filtered was weighed in the container. After ensuring the temperature of the latex at $35 \pm 1^\circ\text{C}$, the container was placed in the position of the MS measuring apparatus. The latex was stirred at the rotational frequency of 1400 ± 200 rev/min throughout the test.

A drop of the latex was removed with a clean glass rod, at every interval of 10 seconds, and gently spread the sample on a water surface and checks the flocculation. The end-point was determines as the first visible flocculation on the water and was confirmed by the presence of increased flocculation in the sample. The time required to initiate visible flocculation was recorded and was expressed the MST as the number of second between beginning of stirring and the end point.

3.2.2. Determination of VFA No:

VFA No of the test samples were determined by ISO techniques published by International Standard Organization. Reference No ISO 506- 1974(E). TSC and DRC of the sample were determined in accordance with ISO 124 and ISO 126.

Five latex samples were tested for VFA No. per one day upon aeration level and altogether forty samples upon maturation.

50(\pm 0.04) g of concentrated latex was taken into a 250ml beaker and accuracy 50ml 30% m/m $(\text{NH}_4)_2\text{SO}_4$ solution was added while stirring with glass rod. The beaker was immersed in 70°C water bath and stirring of latex was continued until it coagulated. Beaker was covered with a watch glass and was left it in the bath for about 15 minutes.



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The serum was filtered through filter paper and the coagulum in the beaker was squeezed with glass rod to get the remaining serum. This serum was filtered through same filter. Then 25ml of filtered serum was pipette out into another conical flask and acidified with 5ml of 50 % Sulphuric acid solution and swirled to mix.

The inner chamber of still apparatus was flushed out with distilled water and 10 ml of acidified serum was added onto it (distillation rate 3-6 ml/min). 100 ml of condense was collected and titrated with standard $\text{Ba}(\text{OH})_2$ (0.01 N \pm 0.001) solution using phenolphthalein as an indicator.

VFA No: was calculated from the following equation,

$$\text{VFA No:} = \left[\frac{134.62 \times C \times V}{m \times \text{TSC}} \right] \times \left[50 + \frac{m(100 - \text{DRC})}{100 \rho} \right]$$

C is the concentration of the barium hydroxide solution, mol/dm³

V is the volume, in cm³, of barium hydroxide required to neutralize the distillate

DRC is the dry rubber content of the latex, expressed as a percentage by mass

M is the mass, in grams, of the test portion

ρ is the density, in g/cm³, of the serum

TSC is the total solid content of the latex, expressed as a percentage by mass

3.2.3 Determination of Alkalinity

Alkalinity of the test samples were determined by ISO techniques published by International Standard Organization. Reference No ISO 125 :1990.

Five latex samples were tested for Alkalinity per one day upon aeration level and altogether forty samples upon maturation.

To 200 cm³ water contained in 400 cm³ beaker add while stirring 10 cm³ stabilizer solution.

Weigh to nearest 10 mg, and add by different from a weighing bottle between 5 to 10 g latex and stir thoroughly.

Inserted the electrode and with continual stirring, add from a burette the hydrochloric acid, until pH is reduced to a value 6.0 ± 0.05 . Add the acid drop by drop on approaching the end point.

As an alternative to electrometric titration, use methyl red as visual indicator, taking the end point as the colour changes from yellow to pink.

Calculate alkalinity as grams of ammonia (NH_3) per 100 g of latex as follows

$$\text{Alkalinity (asNH}_3) = \left[\frac{1.7 \times C \times V}{M} \right]$$

C is the concentration of HCl acid, mol/dm^{-3}

V is the volume required, HCl to neutralized the solution, cm^3

M is the mass of latex taken, in grams

3.2.4 Determination of Viscosity

Five latex samples were tested for Viscosity per one day upon aeration level and altogether forty samples upon maturation.

Approximately 300 ml of latex sample was carefully strained through wire gauze and poured into the glass beaker. Then the spindle bearing No 1 was attached securely to the motor shaft of Brookfield viscometer (Brookfield Syncho- Lectric viscometer). Then the spindle was inserted by placing it vertically into the sample. It was dipped in such a way as to avoid air being trapped and until the surface of the sample was at the mid-point of the groove on the spindle shaft. The latex sample was stirred at the rotational frequency of 30 min^{-1} . Then the viscometer is allowed for rotate about 30 second and stop the dial by using the liver. Viscosity measurement was carried out at room temperature from 10.00 am – 11.00 am. By this method the volume of latex was minimized as much as possible and comparable results were obtained

3.2.5 Determination of Conductivity

Five latex samples were tested for conductivity per one day upon aeration level and altogether forty samples upon maturation.

Conductivity meter was calibrated at 25⁰C using KCl solution as a buffer, having conductivity 1.408 mS when $K=1.00 (\pm 0.005)$. A test portion of approximately 100 ml of latex concentrate from each sample was taken in to plastic containers and it was cooled with gently stirring to 25⁰C using ice water bath (at 23⁰C). From the sample maintained at 25⁰C, approximately 65 ml of latex sample was placed in a plastic container. The probe of the conductivity meter was inserted carefully to avoid inclusion of air bubbles until the surface of the sample is at the specified point on the probe. The probe was gently stirred and equilibrium reading was recorded.

3.2.6 Determination of pH



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Five latex samples were tested for pH per one day upon aeration level and altogether forty samples upon maturation.

pH meter was calibrated at 25⁰C by using pH=4.0 buffer and pH =6.8 buffer. A test portion of approximately 100 ml of latex concentrate from each sample was taken in to plastic containers and it was cooled with gently stirring to 25⁰C using ice water bath (at 23⁰C). When the samples are conditioned the pH probe was dipped in the samples carefully to avoid inclusion of air bubbles until the surface of the sample is at the specified point on the probe. Let the reading stable for few seconds and the equilibrium reading was recorded.