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CREAMING OF PRESERVED FIELD LATEX BEFORE AND AFTER PREVULCANIZATION

Shera Mathew, Roopasri R and Siby Varghese

Abstract

Natural rubber latex collected from the fields of RRII was used in the study. The field latex collected was stabilized and preserved as one percentage ammonia preserved field latex (PFL). This was used to prepare prevulcanized preserved field latex (PPFL). Both the above latices PPFL and PFL was creamed and from this process prevulcanized preserved field creamed latex (PPFCL) and creamed latex (CL) was obtained. The latter CL was also compounded using the same formulation used in PPFL. Thus unvulcanized creamed latex compound (UVCLC) *i.e.* the ordinary creamed latex compound (control) and PPFCL were compared for their processing parameters, physical properties and morphological studies. Cast films prepared from the PPFCL showed better or similar properties when compared to UVCLC. From the above studies it is proved that through this economic method, a product of same or improved quality can be manufactured from this starting material "preserved field latex".

Key words: preserved field latex, creamed latex, processing parameters, control compound

Introduction

Natural rubber latex obtained from *Hevea brasiliensis tree* is a colloid, the dispersed phase is predominantly rubber and the dispersion medium water. The rubber constituent (the elastic component in NRL) which is essentially cis-1, 4- polyisoprene is the main reason for which the rubber tree has been desired for ages. Preserved field latex is unsuitable for most latex applications as its rubber content is low. For most product manufacturing processes, latex with high dry rubber content (DRC) is essential.[1] Latex concentrate is an important raw material with wide range of application. The important methods for concentration of preserved latex are evaporation, creaming, centrifuging, and electro decantation. Concentration by creaming and centrifuging are the important methods of processing latex concentrate. In the above two methods creaming is popular as it often avoids the use of sophisticated tools thus offering a simple and cost-effective route for obtaining latex concentrate.

Creaming is a chemical process involving the addition of creaming agents into the vessels containing field latex to hasten phase separation. It depends on the difference in specific gravity between water (1.0) and rubber polymer (0.91) and can be used for concentrating field latex to higher concentrations. Creaming is due to the formation of a loose network between the molecules of the creaming agent adsorbed upon the surface of the particles and those dissolved in the serum. Buoyancy of the clusters breaks them free from the network

Since specific gravity of rubber particles is less than that of the serum, latex has tendency for creaming. The creaming rate (or settling rate for disperse phases more dense than the continuous phase) can be estimated from the Stokes' equation[2]:

$$\upsilon = 2\, r^2\, (\rho - \rho_o)\, g / 9\eta$$

where, υ is the creaming (settling) rate, r is the diameter of the rubber particle, ρ it's density, ρ_o is the density of the dispersion medium, η is the viscosity of the dispersion medium (continuous phase) and g is the local acceleration due to gravity.

The latex properties of significance to the user are DRC, non-rubber solids (NRS), mechanical stability time (MST), volatile fatty acid number (VFA), potassium hydroxide (KOH) number and alkalinity. Latex concentrate is a non-Newtonian fluid and its viscosity decreases with increasing shear rate.[3] Natural rubber latex has a measurable electrical conductivity due to its salts dissolved in the aqueous phase.[4]

Latex products are manufactured from concentrated latex by mixing it with various compounding ingredients. The compounding ingredients such as vulcanizing agents, accelerators, activators, fillers or any other special additives are solids or liquids and may be water soluble or water insoluble. Preparation of latex compound involves mixing of concentrated natural rubber latex with solutions/dispersions/emulsion of different compounding ingredients. The compounded latex is kept for 24 h for proper maturation. After attaining proper maturation, the products are prepared from the compound. Product preparation process involves steps such as shaping, leaching, drying, beading, vulcanizing, dusting etc. Usually the products can be prepared from the latex compounds directly. In certain cases prevulcanized latices are using for making products like elastic thread, balloons etc. The prevulcanised latex has a similar appearance to that of unvulcanised latex where the former retains the original fluidity, similar latex particles size distribution and stable colloidal characteristic of the latex[5, 6]. Using prevulcanized latex helps to cut short two steps in the routine process. Hence this will be a cost effective process, if preserved field latex can be used directly to make prevulcanized latex for product preparation, the farmers can make the products in their own place.

Considering this requirement, it is necessary to done evaluation study of the products prepared from prevulcanized preserved latex by comparing with the products made from normal procedure. For this purpose in this study the properties of preserved field latex (PFL) & creamed latex (CL) and prevulcanized preserved field creamed latex (PPFCL) & unvulcanized creamed latex compound (UVCLC) were compared, where UVCLC was the normal compounded creamed latex. Finally the products prepared from PPFCL & UVCLC were compared.

2. Experimental Techniques

2.1 Raw materials

The raw materials used are mainly field latex collected from experiment station of the rubber research institute of India. The latex was sieved through a 40 mesh sieve and was ammoniated. Properties of the used latex are given in table 2.1

Table 2.1 properties of used latex

Parameters	Values
Dry rubber content, %	32.6
Non rubber solids , %	3.07
Alkalinity as ammonia, %	1
Volatile fatty acid number	0.04
pH	10.35
Magnesium content, mg/L	258

The details of rubber chemicals and allied materials were Sulphur of Laboratory (LR) grade manufactured by EMERK Ltd., Bombay and Zinc diethyl dithiocarbamate *(ZDC*) of LR grade manufactured by ICI (India) Ltd. Zinc oxide of LR grade manufactured by EMERK Ltd., Bombay was used as activator in this study. It is a coarse white powder, specific gravity 5.47. Ionol (LC) was used as an antioxidant. The above chemicals were made in to 50% dispersions using Dispersol F as the dispersing agent. Other reagents used are summarized in the table 2.2 given below.

Table 2.2 List of other ingredients used

Name of Reagents	Grades used
Ammonia	Commercial grade
Sodium alginate	Commercial grade
Potassium laurate	Prepared in the laboratory from potassium hydroxide and lauric acid
Diammonium hydrogen ortho phosphate (DAHP)	Commercial grade
Acetic acid	Commercial grade
Potassium hydroxide	Reagent grade
Lauric acid	Reagent grade

2.2 Preservation of latex

Sieved field latex (20 kg) collected from the experimental station of the rubber research institute of India, was bulked in a tank and ammoniated to one percentage. 20 kg preserved field latex was mixed with Diammonium hydrogen orthophosphate as 10 percent solution to remove the sludge. After the addition of 25g of DAHP (24 h), removed the sludge from the bottom part of the above preserved field latex. The sludge removed field latex was used for compounding after 15 days of preservation. Then the adequately preserved field latex was divided into 2 batches. One batch of preserved field latex (PFL) latex was compounded and then prevulcanised in a prevulcanization chamber and then creamed. It is notated as PPFCL. The other batch of PFL was kept directly for creaming and after creaming the latex was compounded. It is noted as UVCLC. The formulation used for both these cases were the same, which is given in Table 2.3

Table 2.3 Formulation used for making PPFCL and UVCLC

Ingredients	Parts by weight	
	Dry wt	Wet wt
Field latex	100	313
Potassium hydroxide solution,10%	0.5	5
Sulphur dispersion,50%	1.2	2.4
ZDC dispersion,50%	1	2
ZnO dispersion,50%	0.5	1
Ionol dispersion,50%	1	2

2.3 Prevulcanisation of preserved field latex compound

About 5kg of latex compound in a container was immersed in a water bath set at the required temperature. The compound was subjected to continuous slow stirring and the beaker was kept covered to minimize loss of ammonia. After a predetermined time the compound was immediately cooled to room temperature. On the next day the latex was decanted and filtered through a 100 mesh sieve and stored for further processing.1000g of both prevulcanised latex and preserved field latex was kept for creaming.

2.4 Creaming process

Creamed latex used as control in this study was prepared from the same preserved field latex (PFL). Creaming was done by using sodium alginate as creaming agent. The prevulcanised preserved field latex (PPFL) and the control PFL was treated with 10 percent potassium laurate and sodium alginate and stirred for 30 minutes and was well kept undisturbed for creaming. From the onset of creaming, the TSC of both PPFL and the control PFL was recorded at every 3 hrs. The photographs also have been taken. After 4 days the cream was collected.

Tests such as dry rubber content, ammonia content and magnesium content was done as per IS/ISO specifications for both preserved field latex and creamed latex. The tests such as total solid content, pH, Brookfield viscosity and Mechanical stability time were conducted in latex as well as latex compounds using the relevant specifications.

Tests conducted only with latex compounds (Prevulcanized preserved field creamed latex (PPFCL) and creamed latex compound; here named as unvulcanized creamed latex compound (UVCLC)) were Chloroform test and casting of film. Thin films were prepared by casting the latex compound / prevulcanized latex in shallow glass cells. Strips of glasses were fixed on edges of glass plates (20 cm X 20 cm) to form the cells of size 15 cm X 15 cm. About 15 ml of the latex compound/ prevulcanized latex was poured into the glass cell and distributed so that film of uniform thickness was obtained. The glass cells with the test samples were placed on a leveled table and dried overnight at ambient temperature. The films were removed from the glass cells and subjected to different types of treatment as follows. PPFCL films were dried at 70^0C for 30minutes and UVCLC film were vulcanized at100^0C for 1h. Tests such as equilibrium swelling measurements, ash content, extractable protein (EP) content, stress- strain properties, acetone extraction and morphological studies also were conducted as per relevant specification.

3. Result and Discussion

3.1 *Effect of total solid content (TSC) during creaming*

In latex product manufacturing, latices having high dry rubber content such as concentrated latex, creamed latex *etc.* are generally used[7]. During creaming process the latex get enriched in rubber content and the same time the non-rubber content and other impurities can be separated effectively through the serum. In this study preserved field latex (PFL) was first vulcanized and subjected to creaming. PFL as such (without vulcanization) was used as a reference material. The enrichment in rubber content at the cream portion in both the system was monitored at definite intervals (3h) and is given in figure 3.1 & 3.2. Mechanism of creaming suggested that creaming agent was adsorbed on to the surface of rubber particles and this in some way favors the agglomeration of the particles to form relatively large groups and this enhances the velocity of creaming[8].

In the experiments, it was observed that in both prevulcanised PFL (PPFL) and PFL, TSC was varying with time. The drastic change in TSC was occurred in PFL. But a slow change was observed in the case of prevulcanised PFL which was shown in figure 3.1. The separation of PFL and prevulcanised PFL in to cream and serum can be observed from the optical photographs given in figure 3.2

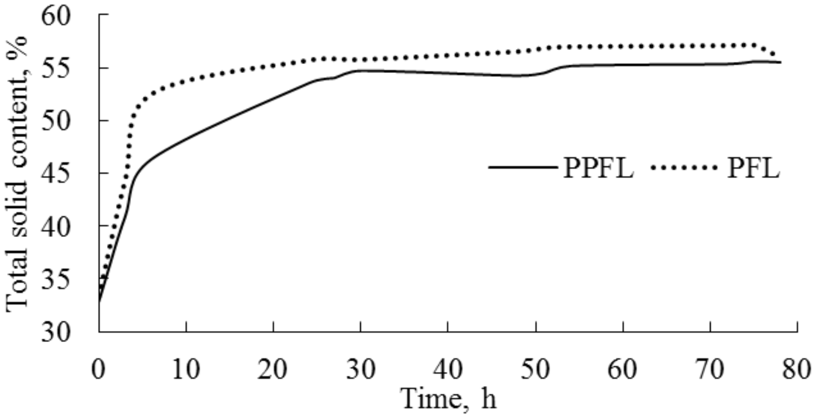

Figure 3.1 variation of rubber content in the cream portion of PFL and prevulcanised PFL

The fact was mainly due to the difference in the chemical constitution of both PPFL and PFL. In the case of PPFL, when latex was prevulcanised, adsorption of compounding ingredients was happened earlier than the adsorption of creaming agent and hence the on-set of creaming of PPFL was slower than PFL. But efficient creaming was observed in the case of PPFL also. But after the adsorption of creaming agents on the surface of prevulcanized rubber particles, velocity of creaming was increased[9, 10]. This was in line with the changes in creaming of both PPFL and PFL.

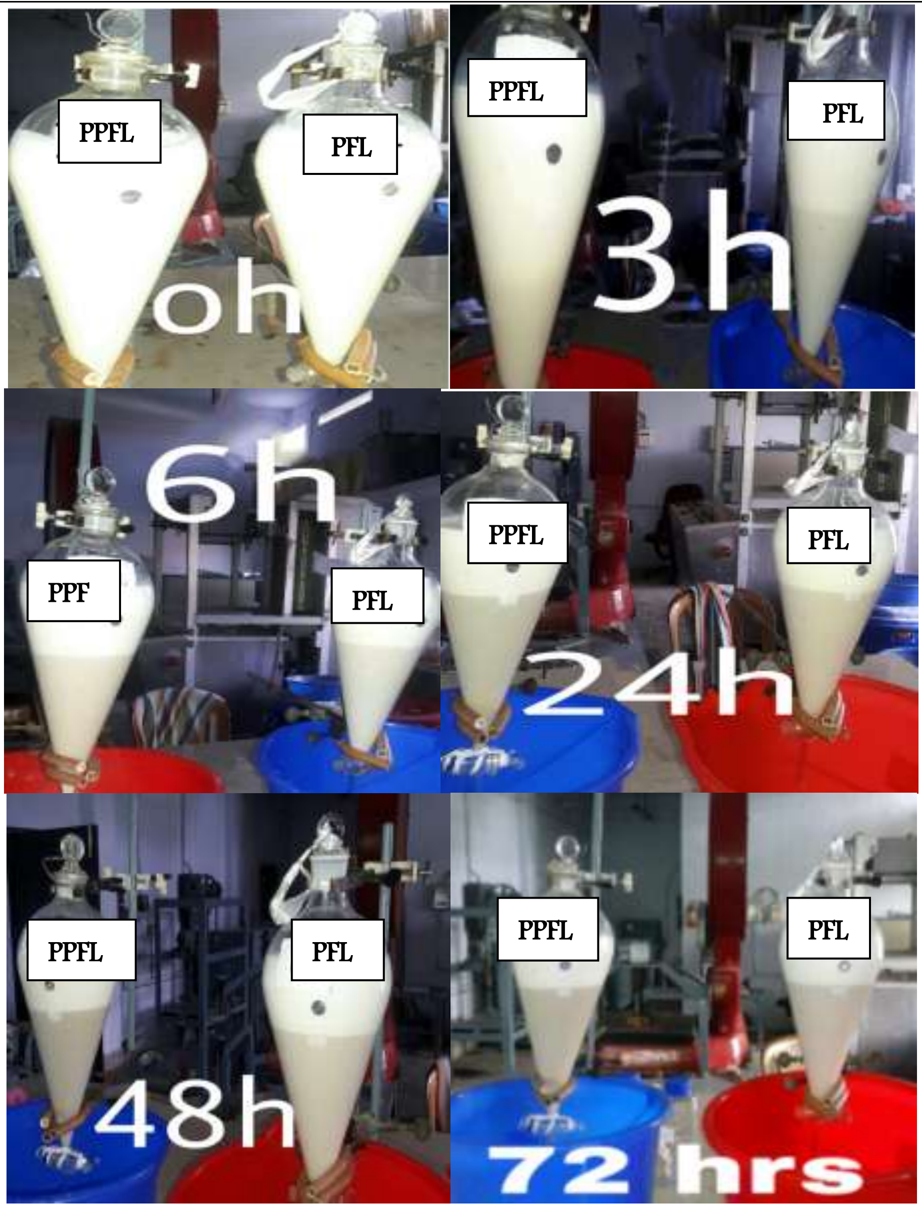

Figure 3.2 Optical photographs showing effect of time on creaming of prevulcanized preserved field latex and (PPFL) and preserved field latex (PFL)

3.2 *Processing parameters*

Processing parameters were tested for latex compounds. For conducting all the tests listed in table 3.1 first total solid content(TSC) was determined and the two compounds was diluted to a total solid of 55 using ammonia water.

The processing parameters tested are given in Table 3.1 below.

3.2.1 *pH*

The effect of pH can be explained on the basis of alkalinity. During prevulcanisation, there is a reduction in ammonia content due to evaporation. At 55^{0}C, the rate of evaporation was higher. Hence pH of prevulcanized preserved field creamed latex (PPFCL) was lower compared with control creamed latex compound (UVCLC).

Table 3.1 Properties of PPFCL and UVCLC (control)

Parameters	PPFCL	UVCLC (control)
Total solid content(TSC), %	55.5	56
pH	10.2	10.4
Brookfield viscosity, cP	207.5	175.5
Mechanical stability time (MST), s	920	836
Particle size, µm	0.52	0.55
Chloroform No	3	2

3.2.2 *Brook field viscosity*

Brookfield viscosity of latex compound depends on its total solid content and particle size distribution. NR latex is a non-Newtonian liquid which exhibits pseudoplastic flow behaviour[8, 11]. During prevulcanisation, crosslinking takes place in every rubber chain[8, 12] of PPFCL than unvulcanized latex compound UVCLC. This crosslinks restricts the movement of the rubber particles; this in turn increases the resistance to flow in PPFCL than UVCLC. Hence viscosity of prevulcanized preserved field creamed latex (PPFCL) was greater than control creamed latex (UVCLC).

3.2.3 *Mechanical stability time*

Changes in the colloidal stability were measured by the mechanical stability time and viscosity[13]. The colloidal stability of prevulcanised latex depends on so many factors, such as the properties of the natural rubber latex, the amounts of potassium hydroxide and carboxylate soap added, the dosage of other vulcanizing ingredients, and prevulcanisation conditions such as the time and temperature[8]. The addition of alkalis and carboxylate soaps will increase the stability by increasing the negative charge on the surface of the particles and by increased surface adsorption. During heating, the presence of alkali may accelerate the hydrolysis of proteins and phospholipids adsorbed on the surface of the rubber particles, there by producing higher fatty acid soap, which increases MST[10]. Hence the MST of PPFCL was higher compared to that of UVCLC.

3.2.5 *Chloroform Number* [10]

Chloroform number is an approximate measure of crosslink density. Determination of chloroform number was done as per the method as described in chapter 2. The coagulum of creamed latex compound (UVCLC) was a weak lump which breaks short when stretched. This indicates a lightly vulcanized state of chloroform number 2. But in the case of PPFCL the coagulum was in the form of non-tacky agglomerates of chloroform number 3. This indicates that PPFCL is at moderately vulcanized state. These can be clear from the photographs given below

PPFCL
Chloroform no 3

UVCLC
Chloroform no 2

Figure 3.3 Photographs showing chloroform numbers of PPFCL and UVCLC compounds

Table 3.2 Properties of PPFCL and UVCLC compounds

Parameters	PPFCL	UVCLC
Swell index	4.2	4.7
Ash content,%	1.2	1.4
Acetone extractables,%	6.2	6.8
Extractable protien content, µg|g		
Before leaching	772	665
After leaching	128.4	138.5

3.3. Property studies conducted using cast films

a) ***Swelling equilibrium***

Swell test is a measure of the crosslink density of films[10]. The swell index values in this study shows that PPFCL is more cross-linked than UVLC. Higher cross links lead to low swell index values this accounts for the observed drop in value in the case of PPFCL compared to UVCLC. Theoretically, when the swelling index is reduced the crosslink density is increased due to denser network chain and restriction in chain mobility. Similar trend could be observed in Nandi and Winter work [14] where with increase of crosslink density, the swelling index is reduced by modeling of poly ethylene irradiated by electron radiation and this also supported by Ruhida et al.[15] where the swelling resistance is increased with increasing crosslink density of NR latex films.

b) ***Ash content***

Ash content is the measure of inorganic substances present in the latex. Greater percentage of ash content was observed in the case of UVCLC compared to PPFCL. This shows that the amount of non-rubber solids (inorganic substances) was higher in UVCLC than PPFCL.

Presence of inorganic substances in the vulcanized film inhibits the light from penetrating through the polymer film. Hence as the non-rubber content increases transparency decreases. This may be the reason for better clarity films derived from PPFCL.

c) *Extractable protein content*

Protein content of PPFCL and UVCLC was compared after and before leaching. Before leaching the protein content of PPFCL film was 772µm|g and that of UVCLC was 665µm|g.There was a slight difference in their values. During leaching water soluble protein were extracted more from PPFCL than UVCLC and which results in lower protein content in PPFCL than UVCLC. The reason for the same may be because of the hydrolysis of protein during prevulcanization may leads to higher protein in the films surface of PPFCL than UVLC and during leaching the water removed the protein in the film surface. The rate of extraction of water soluble materials is faster from prevulcanized films than from post-vulcanized films and increases with the degree of vulcanization[16]. The easier extraction of water soluble materials from prevulcanized latex films is attributed to their higher porosity.

d) *Acetone Extract*

Rubber compounding ingredients like process oils, antioxidants, softness free sulphur etc, (if present) can be extracted with low boiling organic solvents like acetone from an unvulcanized rubber mix or vulcanized rubber products. Acetone extraction shall be considered solely as a quantitative expression of a property normally associated with degree of cure[17]. Properties of rubber mix/articles can be altered by varying the quantity of any one or more of the above components[17]. Changes in the percentage of acetone extractable of PPFCL and UVCLC film (Table 3.2) may be considered as the difference in the degree of vulcanization[18]. As the degree of vulcanization increased, percentage acetone extract will be decreased, here in this study PPFCL was in more vulcanized stage than UVCLC.

3.4 Stress- strain properties

The effect of prevulcanisation of preserved field latex and its creaming on stress–strain properties are shown in the figure below. Before ageing the modulus values were higher in PPFCL than in UVCLC. But after the ageing the modulus of PPFCL was lesser than UVCLC. The high modulus before ageing indicated that maximum crosslinking had taken place at the normal temperature itself. Beyond certain period due to over-vulcanization and the consequent restricted coalescence of rubber particles decrease in modulus was observed (Figure 3.4) [19].

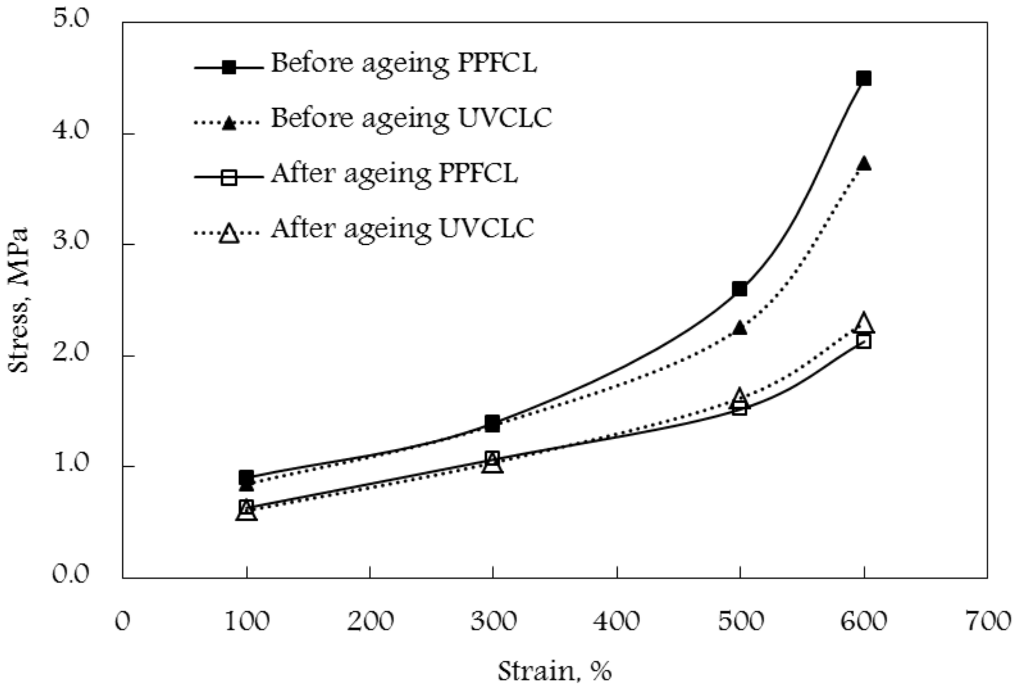

Figure 3.4 Stress-Strain properties of films from prevulcanized preserved creamed latex and the control (Creamed latex compound)

The variation in tensile strength was almost similar to that of modulus. Before giving ageing to the test pieces the tensile strength of PPFCL was higher compared to UVCLC. But after ageing, the tensile strength of PPFCL was lower when comparing with UVCLC. This is mainly because of crosslink shortening and crosslink decomposition. Crosslink decomposition leads to an overall loss of crosslinks which accounts for the observed drop in value.

Table 3.3 Tensile properties of vulcanizates from PPFCL and UVCLC (control)

Test condition	Sample	Tensile strength, MPa	Elongation break, %	Load break, N
Before ageing	PPFCL	21.8	895	49.93
	UVCLC	19.4	896	42.03
After ageing at 70^{0}C for 168h	PPFCL	17.2	929	16.91
	UVCLC	18.0	1015	20.46

3.5 Morphological Study of films

Fig 3.5 shows stereo microscopic photographs of PPFCL and UVCLC films. The figure indicates that UVCLC has a rough structure. Also PPFCL film is more transparent than UVCLC. Prevulcanization of natural rubber latex was a function of maturation duration. The morphology is correlated with the cross-link density of the rubber molecules. Film formed from prevulcanized latex was further postvulcanized, and their film morphologies were compared. Inhomogeneous latex particles cross-linked on the surface with an unvulcanized core were obtained during prevulcanization. This gives rise to the characteristic indentation structure when these hard shell−soft core latex particles coalesce to form film. Basically the prevulcanization mechanism is controlled by the relative rates of the diffusion of vulcanizing reagents and the cross-linking reaction within the latex particles. It is clear that the partially vulcanized particles have a profound influence on the film-forming property of the prevulcanized latex.

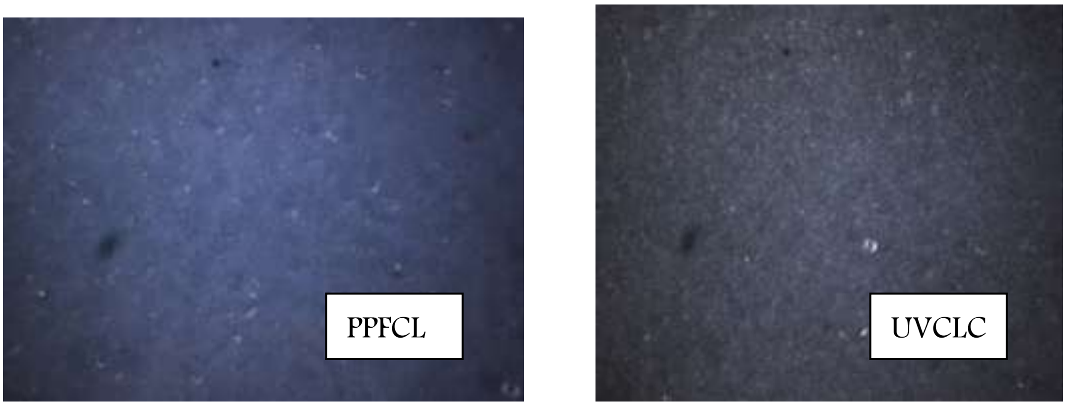

Figure 3.5 a and b Stereo microscopic photographs of PPFCL and UVCLC film

3.6 ***Influence of prevulcanisation on transparency of NR latex films***

One of the advantages with PPFCL was that the sedimental impurities and unreacted ingredients can be removed effectively during creaming which may cause blooming and affect the transparency of the films. But this was not happened in the case of UVCLC film because compounding was occurred after creaming. Removal of these from the prevulcanised latex enables it to form a homogenous film having good clarity. Visual observation also showed that PPFCL film was more transparent than UVCLC film. Morphological investigation was also supported. This was in line with fig 3.5 a and b.

Conclusion

The effect of creaming on prevulcanised preserved field latex (PPFL) and preserved field latex (PFL) were carried out. The effect of total solid content (TSC) during creaming was monitored at regular intervals in both the latices. In this case a comparison has been made with prevulcanised preserved field creamed latex (PPFCL) and creamed latex compound (UVCLC).

In the case of PPFCL and UVCLC the tensile strength and modulus decreased after ageing. This is as expected, because normally in all latex compounds tensile strength was decreases after ageing. However before ageing modulus values were higher in PPFCL than in UVCLC. The maximum modulus values in PPFCL indicated higher level of cross linking.

After a particular period due to over-vulcanization and the consequent restricted coalescence of rubber particles a decrease in modulus was observed.

Ash content of PPFCL was lower compared to UVCLC which indicates the presence of higher non rubber constituents in UVCLC than PPFCL. Extractable protein content is slightly lower than the conventional product after the leaching process.

Morphological investigations confirmed a rough surface for UVCLC film. Transparency of latex film has also been compared. Visual observation showed that PPFCL was more transparent than UVCLC. A substantial increase in transparency is occurred by clarification of prevulcanised preserved field latex preferably by creaming. The optical photos of the films from the two systems were also in support of high clarity of PPFCL film. Thus it is proved that by this economic method, a product of same or improved quality can be manufactured from this starting material “preserved field latex”.

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