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Effect of ammonia preservative systems in centrifuged latex characteristics and latex film properties

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Abstract

Concentrated latex processing facilities are confronted with issues related to high residual phosphate levels in latex. This study was conducted to determine the effects of ammonia preservative systems on residual phosphate levels. Two grades of CL namely, 0.7% ammoniated CL (HACL) and the 0.2% ammoniated CL (LACL) were studied under this survey. Both HACL and LACL samples were characterized for initial total phosphate levels and for other basic latex properties. At the initial stage, the LACL had the higher mean total phosphate level of 434.20 ± 2.20 ppm, while the HACL had the lower level of 412.02 ± 1.42 ppm. Changes in latex properties such as total phosphate levels and soluble magnesium levels of the samples were monitored once a week for a month adhering to ISO standard procedures. A statistically significant interaction between the storage time and the ammonia concentration ($p < 0.05$) on phosphate content was observed. Moreover, the mechanical properties of latex films of both LACL and HACL were studied as well. A statistically significant ($p<0.05$) impact of ammonia concentration on latex film properties was observed. The LACL had the lowest mean for tensile strength (2.49 ± 0.03 MPa), while the HACL had the highest (2.73 ± 0.08 MPa). HACL recorded the highest elongation at break (886 ± 9.31%). In addition, the LACL had the lowest mean for tear strength (8.41 ± 0.01 MPa), while the HACL had the highest (8.78 ± 0.05 MPa). The highest phosphate concentration throughout the storage period was recorded in the LACL sample. Further, the highest mechanical properties were observed in HACL sample over LACL sample. Hence, this study concludes that high phosphate levels impair the mechanical properties of thin films of CL.

***Key words*:** Ammonia preservative system, Centrifuged natural rubber latex, Mechanical properties of latex films, Storage time, Total phosphate content

Introduction

Natural rubber *Hevea brasiliensis* latex (NRL) has a tendency for pre-coagulation on exposure to air, during transit. When rubber particles come into contact with air, proteins that stabilize them degrade to form fatty acids and cause coagulation (Borges *et al.,* 2014). This natural phenomenon known as Pre-coagulation occurs due to the action of bacteria on non-rubber substituents present in the latex. It is evident from the presence of visible clots in the collecting cup and as fermentation bubbles in the coagulum. Non-rubber compounds like, proteins, lipids, and sugars naturally present in the latex are transformed into acids in the presence of bacteria. The development of low molecular weight volatile fatty acids such as acetic and formic acid as a consequence of bacterial activity on carbohydrates cause pre-coagulation, whereas the formation of fatty acids in latex as a result of protein and lipid digestion cause latex stability (Tillekeratne *et al.,* 2003).

Preservatives can be used to prevent pre-coagulation. Proper latex preservation is critical for consistent latex quality characteristics. Anticoagulants such as sodium sulphite, ammonia, and sodium carbonate are used to preserve latex (Riyajan & Santipanusopon, 2010). For concentrated natural rubber latex (CNRL), the preservation method utilized is determined by the end user's specifications. Most commonly used long-term preservatives include ammonia and mixtures of ammonia with a secondary preservative. Secondary preservatives commonly used for this purpose are Tetra Methyl Thiuram Disulphide (TMTD), Zinc oxide (ZnO), and boric acid. Secondary preservatives come in a variety of forms (Tillekeratne *et al.,* 2003). CNRL may be classified into two categories based on the amount of ammonia added in the latex. Low ammonia (LA) latex has not more than 0.29 % ammonia (by weight), and high-ammonia (HA) latex should have not below 0.60 % ammonia (by weight) calculated relative to the latex (Bottier *et al., 1941).*

Due to high pH created by ammonia in latex, bacterial action is prevented. Simultaneously, the phospholipid layer encapsulating the rubber particles too hydrolyze quickly forming glycerol, long-chain carboxylate anions, phosphate, and organic bases over time (Blackley, 1997; Riyajan & Santipanusopon, 2010). The hydrolysis of the phospholipids induces long-term modifications in the chemical composition of the rubber/water interface in latex concentrates (Riyajan & Santipanusopon, 2010). The storage properties of natural rubber latex are also influenced by prominent divalent metal ions such as $Mg^{2+,}$ Ca^{2+}, Fe^{2+}, Cu^{2+}, Mn^{2+} and Zn^{2+} (Zhao *et al.,* 2018). Among them, the presence of magnesium in latex promotes bacterial growth as well, which results in the formation of volatile fatty acids (Tillekeratne *et al.,* 2003), and it has been identified as a key source of flocculation and latex destabilization (Karunanayake & Perera, 2005). To establish a high quality control of concentrated latex, the magnesium content must be eliminated by

introducing phosphate in the form of Di-Ammonium Dihydrogen Phosphate (DAHP) or di-ammonium phosphate into field latex prior to centrifuging leading to the deposition of magnesium (Kaewthai et al., 2019).

$$Mg^{2+}_{(aq)} + (NH_4)_2HPO_{4\,(aq)} \longrightarrow MgNH_4PO_{4(s)} + NH_4^{+}{}_{(aq)} \quad \textit{Equation 1}$$

In addition to the existing natural phosphate levels in centrifuged latex (CL), the addition of excess phosphate concentration is increased in two ways; by accelerating the lipid hydrolysis of ammoniated latex and by incorporating excess DAHP for the removal of magnesium ions from latex.

Ammonia-stabilized latex was later found to accelerate protein and lipid hydrolysis over storage. (Bottier et al., 1941) (Riyajan & Santipanusopon, 2010). So that it will release more phosphate ions into latex over time thereby increasing phosphate level in the latex during storage.

These variations in the phosphate level will have a noticeable influence on the quality of centrifuged latex and on the properties of goods made out of them. It was revealed that an excess of phosphate ions added during processing destabilizes natural latex (Sirikaran, 2012).

Hence, concentrated latex processing plants are confronted with issues relating to latex characteristics such as excess phosphate ions, which reduces the mechanical stability time and increase the KOH number, thereby destabilizing latex compounded for the manufacture of dipped products. Moreover, that will lead to low adhesive properties of compounded latex during dipping process (Sirikaran, 2012). As a result, excess phosphate ions will impair the quality of latex and physical properties of finished products made of them.

Apart from theoretical considerations, no work has been published on the relationship between the phosphate content of centrifuged latex and the preservative system applied. Thus, the purpose of this study was to ascertain the variation in the phosphate content of high ammonia (HA) and low ammonia (LA) centrifuged latex, caused by preservative systems, over storage time as well as its effect on the initial latex properties and latex film characteristics such as tensile strength, elongation at break, and tear strength.

Material and Methods

The study was done at the raw rubber and chemical analysis department of Rubber Research Institute of Sri Lanka (RRISL), Ratmalana. CL samples (from the same source of latex), which were prepared with added HA and LA preservative systems used in this study. Latex samples were purchased from *Lak* Latex Centrifuge (Pvt) Ltd., *Baduraliya.* The *Sigma-Aldrich ACS* grade regents required for latex characterization were used.

Preparation of Samples

HA and LA preserved latex samples, were collected, labelled and chemical treated samples were kept undisturbed for 24 hours at room temperature.

Determination of Initial Latex Properties

After 24 hours, following tests were conducted to assess latex properties of the low ammoniated CL sample (LACL) and high ammoniated CL sample (HACL) following ISO test methods:
Alkalinity by [ISO 125:1990 (E)], Total Solid Content by [ISO 124: 1997 (E)], Dry Rubber Content by [ISO 126:1995 (E)], Volatile fatty acid number (VFA) by [ISO 506:1992 (E)], KOH number by [ISO 127:2008 (EN], Mechanical Stability Time (MST) by [ISO 35:1995 (E), Viscosity by [ISO 1652:1985 (E)], Magnesium ion content by [ISO 17403:2014 (E)], Total phosphate ion content by [ISO 19043:2015 (E)] in order to obtain a comprehensive report of analysis of the 24 hours matured samples.

Determination of Phosphate Ion Content

Determination of phosphate ion concentration of latex was done as per the ISO 19043:2015 (E).

Preparation of Standard Phosphate Solution Series and Calibration curve

containing 0 ppm, 2 ppm, 4 ppm, 6 ppm, 8 ppm, 10 ppm, and 20 ppm, respectively. Then the absorbance of the standard series of solutions was measured with the UV-Vis spectrophotometer at a wavelength of 470 nm. Zero ppm solution was used as the blank. Absorbance was measured in triplicates of each concentration and the average of the three values obtained were taken for comparison. Finally, a calibration curve was drawn by plotting concentrations of standard phosphate solutions against the absorbance.

Preparation of the Latex Samples for Determination of Phosphate Content

20 g ± 0.001 g (w) portions of homogeneous concentrated latex from both HACL and LACL were used in triplicate. Serum of these samples was collected by adding 25 ml of 1:24 HCl solution and placing the sample in a hot water bath at 70 °C for 5 minutes. Then, 10 ml of the serum was filtered and pipetted out 50 ml into a volumetric flask and 10 ml of Vanadium molybdate solution was added to it. Solution was made up to 50 ml adding 1:24 HCl solution.

Determination of Phosphate Content

Absorbance was measured with the UV-Vis spectrophotometer at a wavelength of 470
Nm against the blank (o ppm) solution. The phosphate content of the latex was calculated according to the following equation:

$$P = \frac{[0.05 * Abs * V_{tot} * 3.0661 * 1000]}{S * A * V_{pipette}}$$

Where: P = Phosphate content of the latex sample in milligram (mg), Abs = Absorbance with the spectrophotometer at 470 µm wavelength, V_{tot} = 25+ (W – A) where W is the latex weight in grams, A= [W*TSC]/100, S = Slop of the calibration curve, $V_{pipette}$ = Volume of the serum pipette from the total serum in cubic centimeters (cm^{-3}), 3.0661 = is the constant of conversion of P to phosphate The procedure was carried out as duplicates for both HACL and LACL samples.

Determination of Magnesium Ion Content

Determination of magnesium ion content of latex was conducted as per the ISO 17403:2014 (E). At first, 10 cm^{-3} of serum was pipetted from 10.0 g of HACL and LACL samples separately. After that, to raise the pH up to the range of 10.0 to 10.5, 4 cm^{-3} of NH_4Cl/NH_4OH buffer solution was added. NAHS 1.0 cm^{-3} was added as the masking agent solution. Then a complex-metric titration was carried out with 0.005 $moldm^{-3}$ EDTA solution (standardized with standard magnesium sulfate before the test) using Erichrome Black T (EBT) indicator. The endpoint was recorded when the color changed from violet/red to pure blue. The procedure was carried out in duplicate for both HACL and LACL samples.

Determination of Magnesium and Phosphate Content of CL as a Function of Storage Time

Changes in magnesium ion content and phosphate ion content of the LACL and HACL samples were recorded with storage time once a week, separately, throughout a month according to the aforementioned ISO procedures (Riyajan & Santipanusopon, 2010).

Determination of Latex Film Properties

During the third week of phosphate testing, latex films were prepared by compounding CL according to a basic surgical glove formula (Table 1) approved by RRISL, and the samples were cured in a drying oven at 140°C.

Table 1. *Basic surgical glove formula for latex film preparation*

Ingredients	Parts per Hundred Rubber (PHR)
60% NR latex	100
10% KOH	0.46
10% Lauric acid	0.43
74% Filler (calcium carbonate)	20
50% ZnO	0.5
50% Sulphur	0.9
50% Accelerator	0.6
Water	492.54

Then, the tear strength (ISO 34-1:2011), tensile stress–strain characteristics, and Elongation at Break (EB) (ISO 37-2011) of latex films were evaluated using a universal testing machine.

Results and Discussion

Determination of Initial Latex Properties of CL Samples

Initial physical properties LACL and HACL samples are given in Table 2. Average DRC, TSC, and viscosity of the LACL samples are greater than in the HACL samples, due to the presence of lower non-rubber content compared to those of LACL samples. The initial average VFA of HACL sample was yielded lower values compared to LACL since high ammonia is more effective in suppressing bacterial fermentation (Udayangani, 2013). However, for concentrated latex used for industrial purposes, VFA should be less than 0.05. Hence, from these two parameters both LACL and HACL samples are in acceptable condition. Latex with a high KOH number and a low VFA number are in a higher level of maturity and also that implies a high level of preservation and that is the reason for higher KOH values reported for it than for LACL.

Furthermore, the average initial MST was found to be higher in the case of HACL. These values have a relationship to the phospholipid hydrolysis around the rubber particles because the MST value of them rises with high ammonia levels providing favorable conditions for maximum phospholipid hydrolysis, thereby resulting superior latex stability (Riyajan & Santipanusopon, 2010). Furthermore, in the work done by (Puangmanee, 2016) has mentioned which attribute lower MST values to higher magnesium levels, the observed lower MST levels in the LACL sample can be justified. Moreover, various factors including ammonia concentration, latex concentration, processing parameters, coagulant type, latex composition, stabilizers/additives, and latex age, alongside magnesium levels, can collectively influence the MST in centrifuged latex

Table 2. *Initial latex properties of LACL and HACL samples, (Mean ± SEM of triplicates)*

Latex properties	Latex Samples	
	LACL	HACL
Alkalinity	0.22 ± 0.00	0.74 ± 0.03
DRC	61.25 ± 0.01	60.46 ± 0.05
TSC	62.46 ± 0.03	62.19 ± 0.01
Viscosity (cP)	97.66 ± 0.01	76.33 ± 0.01
VFA No.	0.04 ± 0.02	0.03 ± 0.01
KOH No.	0.61 ± 0.02	0.71 ± 0.01
MST (s)	495.00 ± 0.50	690.00 ± 0.50
Magnesium content (ppm)	144.92 ± 0.10	85.47 ± 0.07
Total Phosphate content (ppm)	434.19 ± 0.55	412.02 ± 0.19
N-RC	1.21 ± 0.00	1.73 ± 0.00

Determination of Phosphate Content over Storage

Total phosphate content of samples matured for one month were recorded for both LACL and HACL samples and they are illustrated in Figure 1. The amount of total phosphate content in both LACL and HACL samples have increased steadily up to the 2nd week (Figure 1), which may be due to the continuous occurrence of fatty acid hydrolysis (Riyajan & Santipanusopon, 2010).

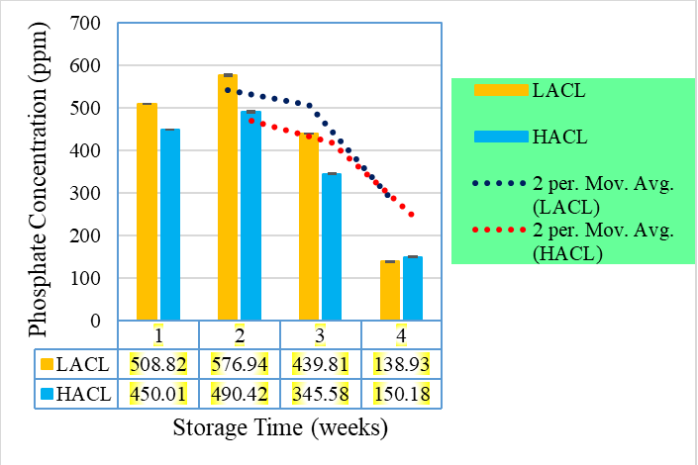

Fig.1. Variation of phosphate content of LACL and HACL samples over storage time. (Mean ± SEM of triplicates)

The peak values of both LACL (576.94 ± 2.80 ppm) and HACL (490.42 ± 4.67 ppm) samples were observed in the 2nd week of storage. Thereafter, a marked reduction of phosphate levels in both LACL and HACL samples was observed. This could be due to phosphate adsorption on the surface of the rubber particle (Jewtragoon, 2004). Either, it could be related to the cessation of lipid hydrolysis or continuous sludge production. In the 4th week of storage, it was observed that, the phosphate content has increased in HACL than LACL which is a deviation comparing to the results of 1st, 2nd, and 3rd weeks of storage. This result can be explained with the synergistic effect of TMTD/ZnO and excess diammonium phosphate in LACL on the gel formation in latex with the storage time. However, total phosphate concentrations in HACL were reported to be lower than in LACL throughout the storage time, which agrees with the statement made by (Riyajan & Santipanusopon, 2010) that gel formation in latex was seen as a result of ionic bonding or the dissociation of phosphate groups by $(NH_4)_2SO_4$. The simple main effect analysis revealed that ammonia concentration and storage time do have a statistically significant effect on phosphate content ($p<0.05$) and this study showed a statistically significant

interaction between the effects of storage time and the ammonia concentration ($p<0.05$) on phosphate content. Overall, the regression was statistically significant (R^2=99.83%).

Determination of Magnesium Content over Storage Time

In the ammoniated medium, continuous phosphate production in the aqueous phase could happen due to the lipid hydrolysis (Blackley, 1966; Jewtragoon, 2004). Hence the sludge production is continued with the produced phosphate and soluble magnesium in the aqueous phase. Therefore, to understand the fluctuation of phosphate level, soluble magnesium content was recorded over a month for LACL and HACL samples and is illustrated in Figure 2.

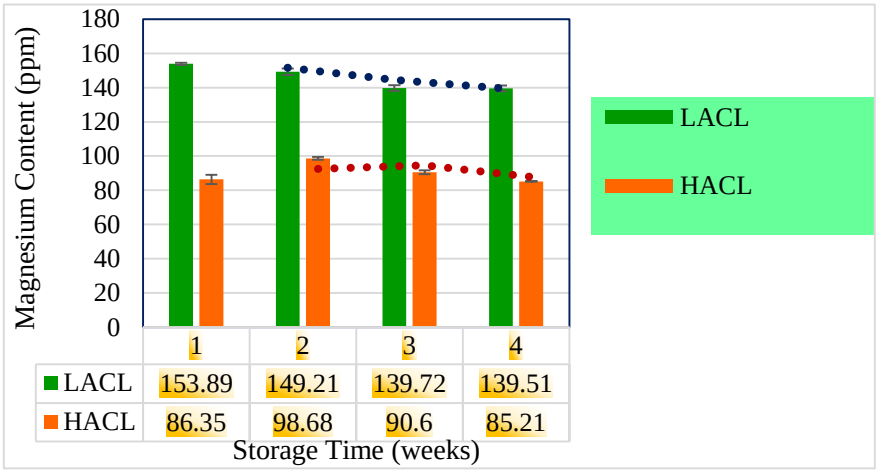

Fig. 2. Variation of the magnesium content of LACL and HACL samples over storage time. (Mean ± SEM of triplicates)

In the LACL sample, a gradual decrement of magnesium levels was observed up to the 4th week. HACL sample was observed with increased magnesium levels up to 2nd week and after that, a gradual decrement was observed.

However, the recorded fluctuations of magnesium levels can be observed due to the action of enzymes that leads to initially or gradually release of magnesium (Jewtragoon, 2004). In overall, it was shown that the amount of soluble magnesium levels reported are lower in HACL compared to LACL throughout the storage time. The similar trend was observed in the work done by (Kaewthai *et al.*, 2019) and stated that it might be the interaction of magnesium ions with ammonia and any orthophosphate anions which are present in the aqueous phase, leading to the

precipitation of the magnesium ammonium phosphate.

There is a statistically significant interaction between the effects of storage content and ammonia concentration ($p < 0.05$) on the resultant magnesium level for LACL and HACL. Overall, the regression was statistically significant ($R^2 = 99.32\%$).

Determination of latex film properties

To understand the impact of ammonia concentration and phosphate levels on the latex film properties of CL, tensile strength, tear strength, and EB were recorded for both samples and presented in Table 3.

Table 3. *LACL and HACL latex film properties after storage. (Mean ± SEM of triplicates)*

Latex Samples (Treatment)	Tensile strength (MPa) ± SD	Tear strength (MPa) ± SD	EB (%) ± SD
LACL	2.49 ± 0.03b	8.41 ± 0.01b	769.73 ± 10.20b
HACL	2.73 ± 0.08a	8.78 ± 0.05a	886.00 ± 9.31a

Means that do not follow the same letter within each column are significantly different at $p < 0.05$.

Non-rubber components of natural rubber latex, primary proteins and mono-or di-phosphates, assist in the crosslinking process and boost the crosslinking density and tensile strength of vulcanized latex films (Wei *et al.,* 2020) (Sriring *et al.,* 2018). Moreover, ammonia also can cause the development of cross-links in rubber vulcanizate (Yu *et al.,* 2015). Hence, higher tensile strength in HACL indicates the higher cross-link density, which correlates to higher tear strength in HACL than in LACL sample. However, tensile strength of the LACL and HACL samples are quite low in comparison to the average value (24 MPa) for a glove compound according to ASTM specifications (Ibrahim *et al.*, 2003). These results can be explained by the reduction of gel formation over time occurring due to cleavage or denaturation of rubber proteins via enzymatic processes in latex (Nawamawat *et al.,* 2010).

In addition, when NRL films are produced, the filler is intended to adhere to the rubber particles like other non-rubber materials. This is similar to natural surface materials (Roslim and Hashim, 2010).According to the used formula for latex film preparation, calcium carbonate has been used 74%, which is a significant amount (more solid foam) that may have an impact on the appropriate dispersion of filler through the rubber matrix. The compatibility effect within the film may be reduced by the formation of massive calcium carbonate clusters in the films. At the same time, it can cause localized stress in weak spots, which would lead to poor physical properties. This can be further supported by looking at the NRL surface

morphology in relation to the addition of fillers that were captured using a SEM in later research.

Furthermore, it is well known that fillers merely make films stiffer; they have no positive effect on cross linking (Roslim and Hashim, 2010). It appears that the filler's stiffness alone may be the cause of the reported strength values. The strength of NRL films cannot be increased by filler alone because of the low sulphur concentration in latex films.

It was shown that the EB of both latex film samples exceeds the minimal range of ASTM standards (400 to 750%), as shown in Table 3. Having proper EB values in both LACL and HACL samples are important because the gel formation is strongly related to latex characteristics. Average reduction of crosslinking density/gel formation is inversely related to the EB (Nasruddin & Susanto, 2020; Sriring et al., 2018).

Conclusions

In general, LACL retains a significantly higher total phosphate concentration at both onset and throughout the storage time. However, both samples gradually increase their phosphate content as a function of storage time up to the 2nd week. Thereafter, it will fall steadily to the 4th week, indicating the interaction of poly-isoprene chains with phosphate groups present in the latex. Moreover, the lower soluble magnesium levels reported in HACL can happen due to the precipitation of the magnesium ammonium phosphate which reduces the soluble magnesium in the aqueous medium of latex. The LACL sample displays significantly lower mean values for all the mechanical properties tested. However, the HACL shows highest values for each properties; tensile, tear, and elongation at break, respectively. However, both samples showed EB values that are greater than 750%. Latex stability is strongly related to the latex characteristics. Hence, one could argue that the stability of CL relies on the storage time with varied ammonia content. Finally, this study evidences that there is a significant influence of ammonia concentration on phosphate content and the characteristics of latex film properties.

Moreover, it is evident that excess phosphate ions will impair the quality of latex and end products of rubber. However, further research needs to be done to examine more about the chemical structural level influence of ammonia and other primary preservative systems on the phosphate content of CL and on the overall anionic concentration of latex.

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