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Research Article

Effects of Ammonium Laurate Concentration on Physico-mechanical Properties of Natural Rubber Latex

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Abstract

Natural rubber latex tends to coagulate and putrefy shortly after harvesting. Therefore, ammonium laurate is added to stabilize natural rubber latex. However, excessive level of ammonium laurate may adversely affect physico-mechanical properties of natural rubber latex. This study was carried out to investigate the effects of ammonium laurate concentration on physico-mechanical properties of natural rubber latex with special reference to the glove formulation. Partially preserved, concentrated rubber latex samples were treated with varying amounts (0, 2, 4, and 8 ml) of 10% (w/v) ammonium laurate solution. Potassium hydroxide number, volatile fatty acid number and mechanical stability time of the samples were investigated weekly for a period of one month. After 21 days of maturation of latex samples, films were prepared and their tensile strength, elongation at break and tear strength were investigated. Ammonium laurate concentration significantly ($P<0.05$) affected on both mechanical stability time and volatile fatty acid number of latex during the storage time. The highest tensile strength and elongation at break were observed in the sample treated with 1.5×10^{-4} moles of ammonium laurate per 100 g of latex. Tear strength showed a positive linear relationship with the ammonium laurate concentration. It can be concluded that the ammonium laurate level of 1.63×10^{-4} moles per 100 g of latex will enhance the physico-mechanical properties of natural rubber latex without negatively affecting its chemical properties such as VFA number and KOH number.

Keywords: *Ammonium laurate, mechanical stability, potassium hydroxide number, volatile fatty acid, tensile strength*

1. Introduction

Bio-surfactants stabilize natural rubber latex (NRL) by exerting electrostatic repulsive forces between rubber particles. However, the phospholipid-protein complexes of latex are usually destroyed by bacteria. Synthetic surfactants are added to minimize the loss of stability of rubber particles caused by the destruction of phospholipid-protein complex (Kasan *et al.*, 2015). The hydrophilic part of the surfactant molecules can interact with the medium. Surfactants adsorbed on the surface of the rubber particles exert electrostatic repulsion between rubber particles preventing further flocculation (Kasan *et al.*, 2015). Surfactant molecules present at the interface between the rubber phase and the aqueous phase potentially affect the interfacial tension and thereby the particle morphology. Maturation allows the system to become homogeneous in its composition. Various changes can occur in the latex system during the "maturation" process. Fatty acids have been known to influence the membrane surrounding the rubber particle. Many efforts have been made to investigate the effect of fatty acids soaps on the mechanical stability of latex (Blackley, 1999). Previously reported results revealed that the addition of small amounts of ammonium laurate could considerably enhance the mechanical stability time (MST) of the latex. Although fatty acids can increase the mechanical stability of latex, they can cause some problems in latex such as foaming, which results in poor properties such as thin walls and pinholes in dipped products. However, explicit data on variation of physico-mechanical properties such as tensile strength along with elongation at break and tear strength of natural rubber latex with the concentration of ammonium laurate are rarely available in the literature.

Therefore, this study was undertaken to investigate the effects of ammonium laurate concentration on tensile strength, elongation at the break, and tear strength of natural rubber latex compound and to determine the optimum concentration of ammonium laurate for preparation of glove compound.

2. Materials and Method

2.1. Experimental design

Treatments were assigned completely at random and the experiment was carried out in triplicate.

2.2. Materials

Partially preserved (without adding Tetramethylthiuram disulfide/ZnO during centrifugation) concentrated natural rubber latex was collected from a leading latex manufacturer in Sri Lanka.

General-purpose ammonium hydroxide and commercial-grade lauric acid were used and all other chemicals used in this study were of analytical grade. All the chemicals used in this study were available at the Department of Raw Rubber and Chemical Analysis, Rubber Research Institute of Sri Lanka.

2.3. Preparation of 10% (w/v) ammonium laurate solution

Aqueous ammonium laurate 10% (w/v) solution with pH of 10.0 was prepared by neutralizing ammonium hydroxide with lauric acid.

2.4. Preparation of natural rubber latex samples with different Ammonium laurate levels

The collected latex sample was strained through a wire mesh to remove any coagulum. Four latex samples (four liter each) were taken into four plastic containers with the same capacity (6 L). Different amount of 10% (w/v) ammonium laurate was added to each sample as mentioned in the Table 1. Then the samples were stirred for 2 minutes. After closing the lids of the containers, these samples were stored at room temperature of 29 ± 1°C for 24 hours to allow equilibrium.

Table 1: Treatments

Sample Code	Latex volume (L)	Amount of 10% (w/v) ammonium laurate added (mL)	Ammonium laurate concentration (moles/100 g of NRL)
A	4	0	0
B	4	2	1.5×10^{-4}
C	4	4	4.2×10^{-4}
D	4	8	5.0×10^{-4}

2.5. Preparation of natural rubber latex films

After 21 days of sample maturation, latex compound was prepared according to a surgical glove formula. Dispersions were made by making coarse slurries of powdered ingredients in water with dispersing agents and then by grinding to average particle size of about 5 micron. Finally, glove compound was made by adding ingredients to a mixing cycle and stirring for 12 minutes. The prepared compound was poured into glass molds for film casting. The molded compound films were firstly, air-dried overnight, and then oven-dried at 60°C for 30 minutes.

2.6. Determination of chemical and physico-mechanical properties of initial latex samples

Alkalinity, potassium hydroxide number (KOH), volatile fatty acid number (VFA), total solid content (TSC), dry rubber content (DRC), viscosity, mechanical stability time (MST) of the prepared latex samples were determined in triplicate using the following methods.

Alkalinity was determined as concentration of ammonia (%) according to the ISO 125: 1990(E) method by titrating diluted latex sample with 0.05 $moldm^{-3}$ sulfuric acid in the presence of bromothymol blue indicator whereas KOH number was determined by performing potentiometric titration using 0.5 $moldm^{-3}$ KOH solution as titrant (ISO 127: 2018 (E)). Serum was separated from a known weight of latex and it was subjected to distillation and volatile fatty acid number was determined by titrating the distillate with a 0.005 $moldm^{-3}$ $Ba(OH)_2$ solution in the presence of phenolphthalein as an indicator (ISO 506-1992(E)). Total solid content (%) was determined by drying a known weight of sample (2.0000 g) in an oven at 105 °C for about 2 hours (ISO 124-1977(E)). For determination of dry rubber content, a known weight of the latex sample was coagulated with 0.2% acetic acid, pressed to a sheet, washed and weighed after drying in an oven at 70°C (ISO 126-1995(E)). Viscosity was determined following the ISO 1652-1985(E) method using a viscometer (Brookfield) and mechanical stability time was determined according to the ISO 35-1995(E) method using a mechanical stability Tester (Kalaxon).

2.7. Determination of chemical and physico-mechanical properties of initial latex samples during the storage

KOH number, VFA number and MST of the prepared latex samples were determined weekly for a period of one month using the above methods (2.6). These analyses were carried out in triplicate.

2.8. Characterization of latex film properties

Tensile strength along with elongation at break and tear strength of the latex films were determined in triplicate. Tensile strength along with elongation at break was determined in accordance with ISO 37-2011 method using a universal tensometer whereas tear strength of latex films was measured in accordance with ISO 34-1: 2015 (E) method using an universal tear testing machine.

2.9. Data analysis

Data were subjected to Analysis of variance (ANOVA) and Tukey pairwise comparison using Minitab 17 software.

3. Results and Discussion

3.1. Effects of ammonium laurate concentration on initial properties of the natural rubber latex

Alkalinities of all samples were below the maxim stipulated level of 0.29% for centrifuged low-ammonia preserved latex (ISO 2004: 2017). Further, alkalinity levels of all samples were not significantly different (Table 1). Concentration of ammonia is crucial for adequate protection of the latex against microorganisms and for maintaining the colloidal stability (Kędzia et al., 2010).

Table 1: Initial properties of the prepared latex samples

Latex properties	Samples (ammonium laurate concentration in moles/100 g of latex) *			
	A (0)	B (1.5×10^{-4})	C (4.2×10^{-4})	D (5×10^{-4})
Alkalinity (%)	0.24 ±0.01[a]	0.25 ± 0.01[a]	0.24 ± 0.01[a]	0.25 ± 0.01[a]
KOH number	0.42 ± 0.00[c]	0.47 ± 0.00[b]	0.48 ± 0.00[ab]	0.49 ± 0.00[a]
VFA number	0.014 ± 0.0[d]	0.021 ± 0.0[a]	0.014 ± 0.0[d]	0.018 ± 0.0[b]
TSC (%)	62.3 ± 0.0[a]	61.4 ± 0.1[a]	61.8 ± 0.1[a]	62.1 ± 0.0[a]
DRC (%)	60.8 ± 0.0[a]	60.2 ± 0.0[a]	60.8 ± 0.0[a]	60.6 ± 0.0[a]
Viscosity (cP)	100.0 ± 0.0[a]	83.3 ± 0.3[b]	82.16 ± 0.2[b]	80.00 ± 0.6[b]
MST (s)	87.6 ± 0.2[c]	306.0 ± 0.6[b]	1800 ± 1[a]	1800 ± 1[a]

*Figures are mean ± Standard Deviation (n=3); Figures with different superscript letters in a raw differ significantly ($p \leq 0.05$); KOH number (as number of grams of KOH equivalent to the acid radicals combined with ammonia in latex containing 100 g of total solids); VFA-volatile fatty acid number (as number of g of KOH equivalent to the volatile fatty acid in the latex containing 100 g of total solids); TSC-total solid content; DRC-dry rubber content; MST-mechanical stability time.

KOH number is defined as the number of grams of potassium hydroxide equivalent to all the acid radicals combined with ammonia in a quantity of latex-containing 100 g of total solids. KOH numbers of all samples were below the maxim stipulated level of 0.70 for centrifuged low-ammonia preserved latex (ISO 2004: 2017). Initial KOH number showed a positive linear relationship with the ammonium laurate concentration in latex samples (Figure 1). Ammonium laurate can increase the total ion concentration of the medium because it is an ionic surfactant (Akhtar *et al*., 1995) and also it can increase the ion concentration of the medium by hydrolyzing lipid of the latex particles (Sansatsadeekul *et al*., 2011). KOH number increased with the concentration of ammonium laurate (Table 1) because ion concentration in the medium positively affect the KOH number (Pendle, 1990). Among the anions present in latex, some like fatty acids and protein can serve as stabilizers whereas, the others like volatile fatty acids, phosphate, carbonate etc. can decrease the stability. Therefore, for better explanation, KOH number result should be related to other parameters too. KOH number between 0.3- 0.5 results in good latex qualities (Jordan, 1939). According to the results, ammonium laurate concentration between 1.5×10^{-4} - 4.2×10^{-4}

moles per 100 g of latex showed acceptable KOH number together with other tested two parameters of natural rubber latex (Figures 2 and 3). Nevertheless, an ammonium laurate concentration of 1.63 × 10^{-4} resulted in optimum physico-mechanical properties without negatively affecting the chemical properties of natural rubber latex.

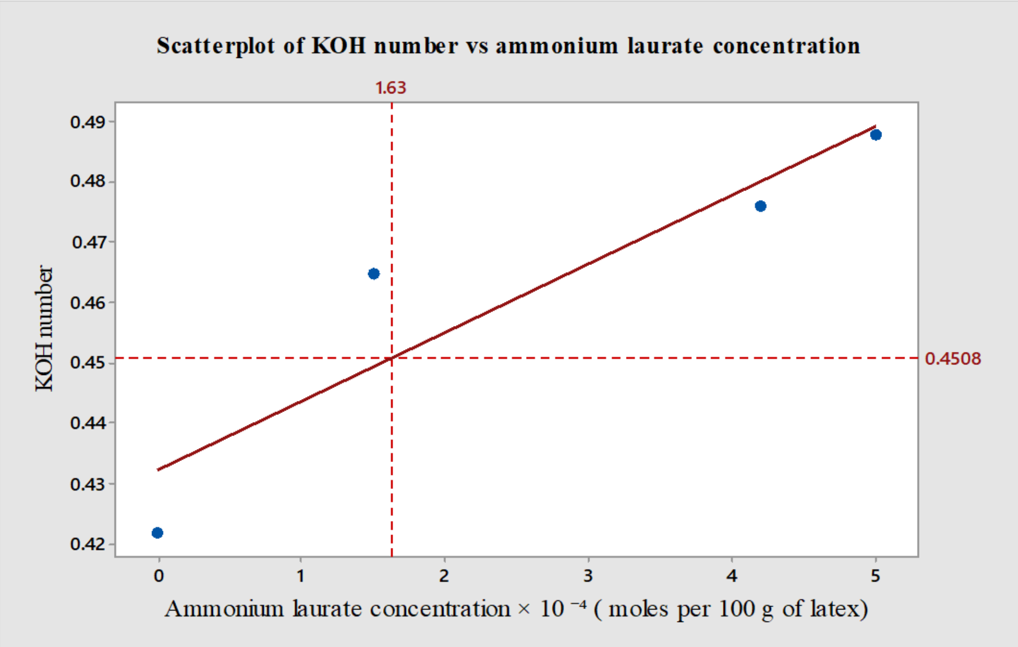

Figure 1: Scatterplot between KOH number and ammonium laurate concentration

VFA number describes the number of grams of barium hydroxide ($Ba(OH)_2$) equivalent to the steam distillable fatty acids present in a quantity of latex-containing 100 g of total solids. VFA numbers of all samples remained below the maximum stipulated limit of 0.06 (Table 1). Presence of non-rubbers in the latex has the ability to produce volatile fatty acids via hydrolysis under ambient temperature (Riyajan *et al*., 2010) increasing the VFA number. The initial VFA has a positive relationship with the non-rubber content of the latex.

Total solid content is the mass percentage of both suspended and dissolved solid materials present in latex. As shown in the Table 1, total solid content of all the samples were above the minimum requirement (61%) stipulated for centrifuged low-ammonia preserved latex (ISO 2004: 2017) and total solid content was not affected by ammonium laurate concentration.

Dry rubber content is the percentage of dry rubber materials present in the latex. Further, dry rubber contents of all four samples were also over the minimum requirement of 60% (ISO2004:2017) and it was not affected by ammonium laurate concentration.

Viscosity is the resistance of latex to flow. Viscosity of latex has great impact on the processability. Nevertheless, it is not specified in ISO 2004 due to varying customer preferences. The results showed that viscosity of latex samples reduced with the addition of ammonium laurate (Table 1). This reduction is due to the distortion of the original structure in the solution by ammonium laurate (Verhaar, 1959). Further, randomly adsorbed foreign ammonium laurate molecules result in displacing protein on the surface of the rubber particle changing its shape and size and their by the viscosity (Verhaar, 1959).

The mechanical stability time (MST) of latex is the duration of time in seconds taken to reach latex instability (visible sign of coagulation occurs) when the latex is stirred at a specified rotational frequency (14,000 rpm). MST of latex indicates the colloidal stability of concentrated natural rubber latex (CNRL). MSTs of the control sample and the sample treated with 1.5×10^{-4} of ammonium laurate (moles/100 g of NRL) were below the minimum requirement of 650 (s) stipulated for centrifuged low-ammonia preserved latex (ISO2004:2017). However, with the addition of ammonium laurate, MST increased (Table 1). This is a result of stabilizing latex due to electrostatic repulsive forces exerted by ammonium laurate (Pendle, 1990).

3.2. Variation in properties of latex samples during the storage

3.2.1. Potassium hydroxide number

Figure 2 illustrates the variation in KOH number of samples with the storage time. KOH number increased with the storage time presumably due to many acid radical formations (Kędzia *et al*., 2010). Therefore, it can be an indicator of age of latex. Many free acids, amino acids, oxalic acid, phosphoric acid, and succinic acid have been reported to be present in latex serum (Sansatsadeekul *et al*., 2011). Initially, KOH number increased at higher rate and later, it increased at lower rate. Increase in ion concentration of the medium due to hydrolyzing lipids of the latex particles by ammonium laurate resulted rapid increment of KOH number at the initial stage (Blackley, 1997; Sansatsadeekul *et al*., 2011). Ammonium laurate may be found in the medium in monomolecular or micellar forms, when they are ionized. Ion strength of the aqueous phase equalized with added ammonium laurate anions and cations. When the latex medium is saturated, the excess ammonium laurate molecules present in the medium tend to aggregate in micelles (Ng *et al.*, 2022). It limits the further ionization of the molecules and the medium becomes saturated with time (Gao, 2019). This suggests that the KOH number becomes slightly constant with time (Arnold *et al.*, 1987). This leads to the conclusion that ammonium laurate molecules in the ionized form in the medium are complementary to the KOH number.

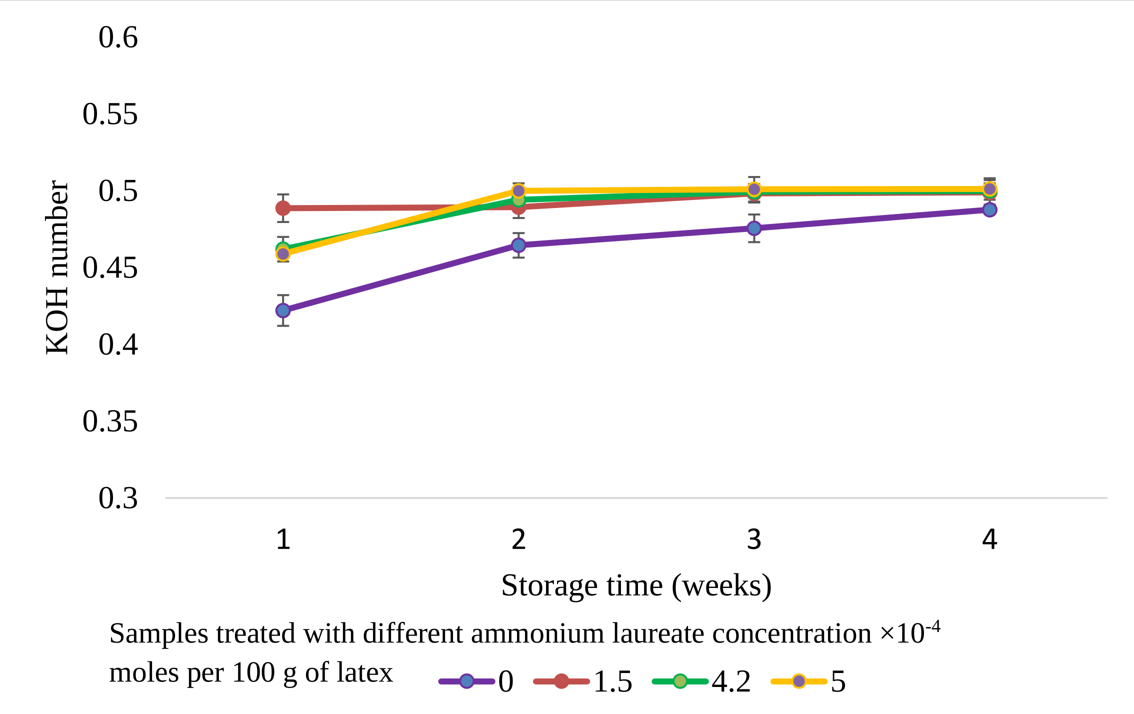

Figure 2: Variation in KOH number of latex samples during the storage time

3.2.2. Volatile Fatty Acid number

Variation in VFA number of the latex samples during the storage period is shown in Figure 3. For good quality latex, VFA number should be within 0.02- 0.03 (Venkatachalam *et al.*, 2013). Non-rubber materials present in latex can support bacterial growth and produce volatile acids such as formic acids acetic acid, etc. (Riyajan *et al.*, 2010). Ammonium laurate, up to a certain concentration, has potential to increase antimicrobial properties (Sarih *et al.*, 2022). However, ammonium laurate is not capable enough to control the lipid hydrolysis of non-rubbers (Somarathna *et al*., 2018) and therefore, as observed, VFA number gradually increased again after 21 days of maturation.

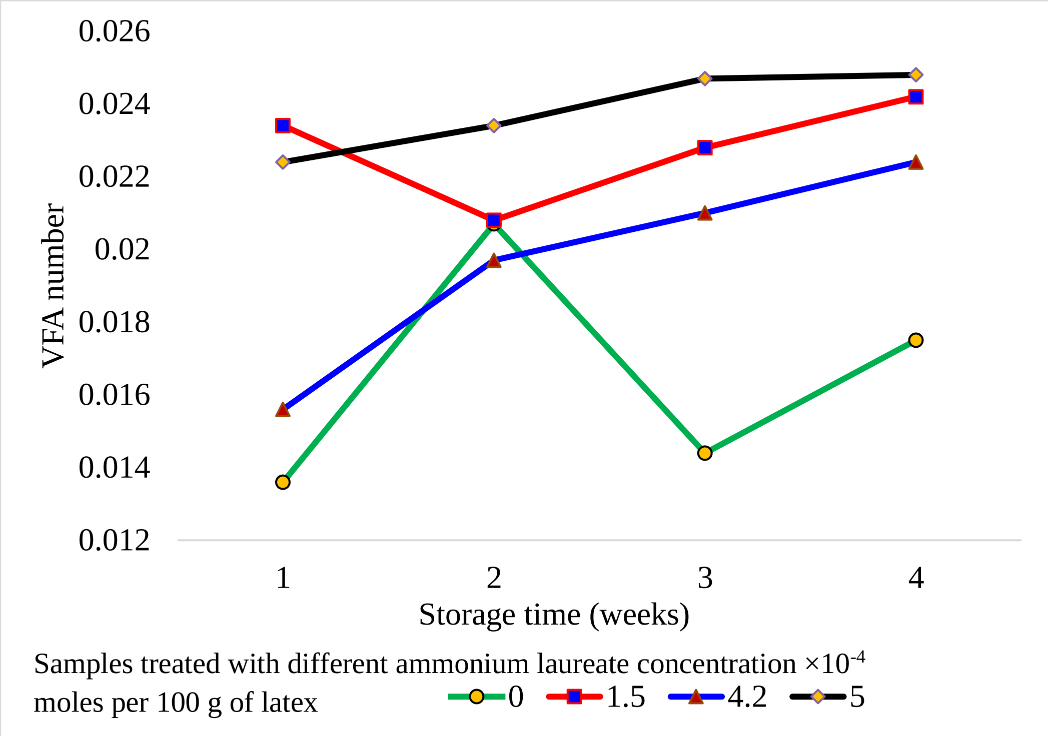

Figure 3: Variation in VFA number of samples during the storage.

3.2.3. Mechanical stability time

Results showed that very small amount of ammonium laurate could produce significant enhancement in the mechanical stability of low-quality NRL compared to the control (Figure 4). Nevertheless, ammonium laurate concentration above 4.2 $\times 10^{-4}$ moles/100 g of latex did not result significant difference in MST. This may be due to the attainment of critical micelle concentration (CMC) of soap in the medium. Ammonium laurate is a poor mechanical stabilizer when compared to fatty acid soaps (Pendle, 1990). By displacing some proteins, ammonium laurate adsorbed at the interface and mixed with adsorbed fatty acid anions. Its sufficiently short chain length may have disrupted and dispersed the coherence of clusters of indigenous soap anions. Mixing of indigenous soap islands increases repulsive energy between particles. This happened by clearing the way to approach hindered weak proteins and their displacement. This phenomenon may be a dominating factor in enhancing MST of samples. The nonpolar–nonpolar interaction of the latex sample does not provide the required thermodynamic energy for micelle formation. However, the ionic surfactant, ammonium laurate has a higher CMC value. It reduces the surface tension of the latex samples while increasing the latex particle movements (Kondo *et al.*, 2007). MST between 600 -1000s is considered as the best for most of the applications.

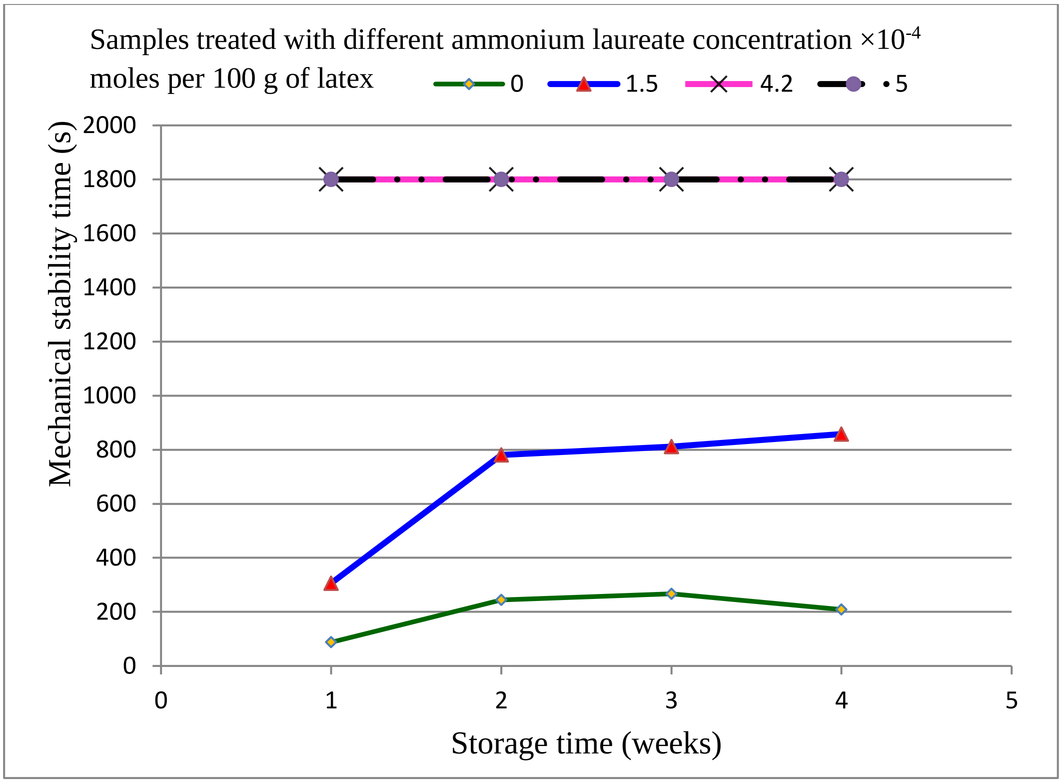

Figure 4: Variation in MST of latex samples during the storage.

3.3. Effects of level of ammonium laurate concentration on physico-mechanical properties of latex films

3.3.1 Tensile strength

Figure 5 illustrates the changes in tensile strength of latex films with the added ammonium laurate concentration. Ammonium laurate surfactant would efficiently reduce and remove proteins from natural rubber latex particles through denaturation by breaking the protein-protein interactions and transferring them to the serum phase. Denaturation involves the breaking of many of the weak linkages or bonds within a protein molecule that are responsible for the highly ordered structure of the protein in its natural state (Pailhories, 1993). Protein denaturation affects the total nitrogen content of the natural rubber latex films (Nawamawat *et al*., 2010). This nitrogen and added sulphur during film preparation allow crosslinking (Wei *et al*., 2022). However ammonium laurate inhibits crosslinking to a certain extent due to particle's repulsive forces ((Nabzar *et al.*, 1998; Maznah *et al*., 2008). Reduction in crosslinks results in lower tensile strength due to the lower number of linkages. Saponification also capable of reducing the tensile strength of the latex (Nawamawat *et al*., 2010). Therefore, tensile strength shows a negative linear relationship with ammonium laurate concentration (Nawamawath *et al*., 2010). All the samples had lower tensile strength than the recommended level (14 MPa) for glove compound (Buritatum *et al*., 2022). Usage of low-quality natural rubber latex results in this. Partial preservation limits the zinc oxide amount present in the latex, which has a great impact on tensile strength enhancement (Shi, 2001). However, the tensile strength of the sample treated with 1.5 x 10^{-4} moles of ammonium laurate per 100 g of latex (sample B) was higher than that of other samples. This may be due to the increment of crosslinks in the point up to 75% gelation occurs (Lee, 2005).

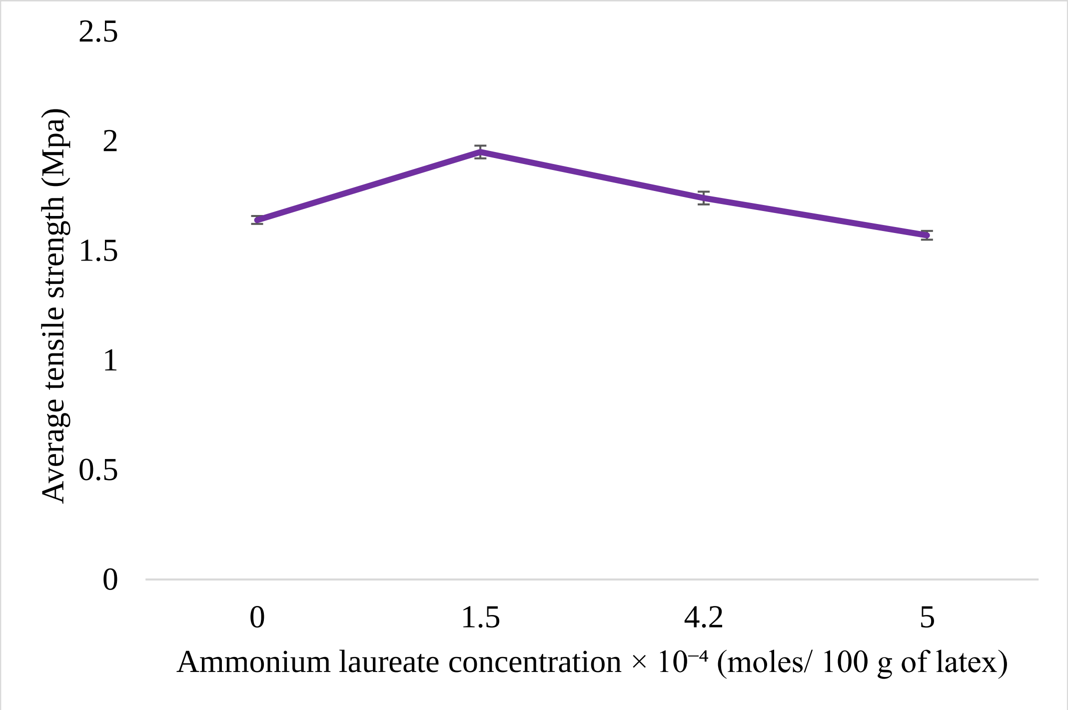

Figure 5: Effect of ammonium laureate concentration on tensile strength of latex films.

3.3.2. Elongation at break

According to the ASTM D 412 standards, the expected elongation at break should be within 400-750%. Crosslinking density has a negative relationship with the elongation at the break of the latex films (Lal, 1970). Variation of elongation at the break of the latex films prepared with different ammonium laurate concentration is shown in Figure 6. The higher reported values of elongation at break may be due to the lower crosslinks as a result of the high mobilization of latex particles (Nasruddin and Susanto, 2020). Formation of a three-dimensional network by chemical or physical cross-linking which is referred to as gel formation (Rochas and Rinaudo, 1984) also results in higher values of elongation at break. The highest elongation at break showed by the sample treated with 1.5 x 10^{-4} moles of ammonium laurate per 100 g of latex (sample B) because of the increment of crosslinks in the point up to 75% gelation occurs (Lee, 2005).

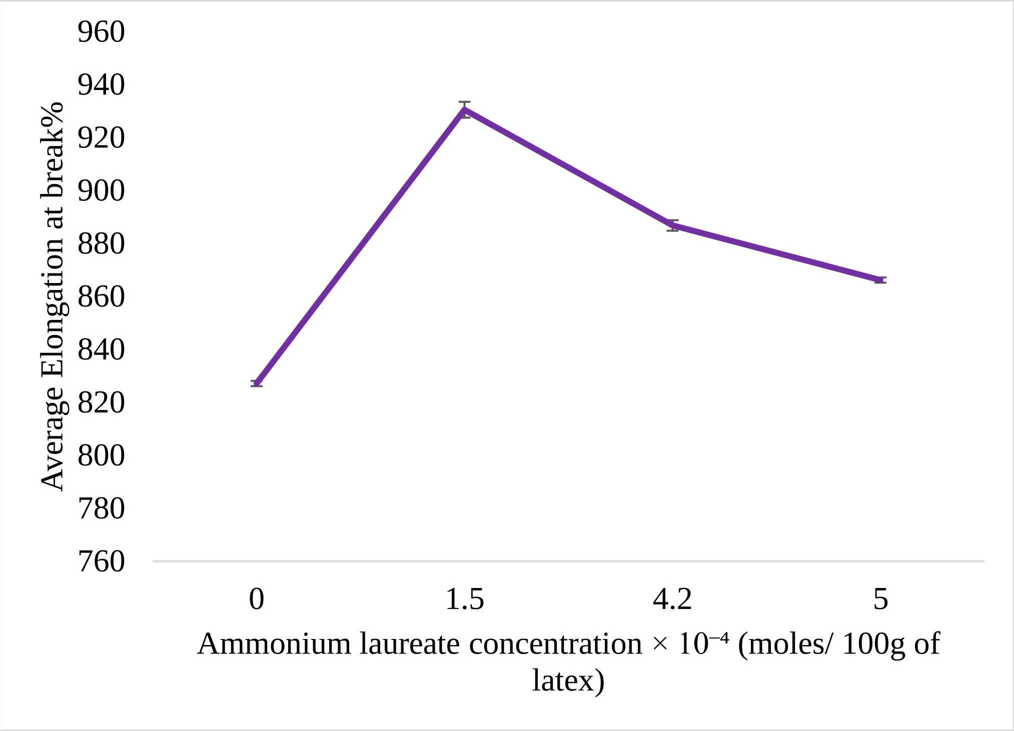

Figure 6: Effect of ammonium laureate concentration on elongation at break of latex films

3.3.3. Tear strength

Tear strength is defined as the maximum force required to tear a test specimen in a direction normal to the direction of the stress. Stabilizers had a significant effect on the tear strength of the latex (Sutanto *et al*., 2014). Ammonium laurate stabilizer helps in enhancing the KOH number of the latex by increasing total ion concentration (Silva and Walpalage, 2009). The KOH number has a significant effect on the tear strength of latex films (Sin *et al.*, 2015). The maximum ion concentration in the medium showed after 21 days of maturation because the medium becomes equilibrium. The highest tear strength showed by the sample treated with 5 x 10^{-4} moles of ammonium laurate per 100 g of latex (sample D). This can be due to the higher ion concentration of the medium. The expected tear should exceed the value of 14 MPa, partial preservation of the latex limits tear to the lower values due to lack of zinc oxide accelerator (Shi, 2001).

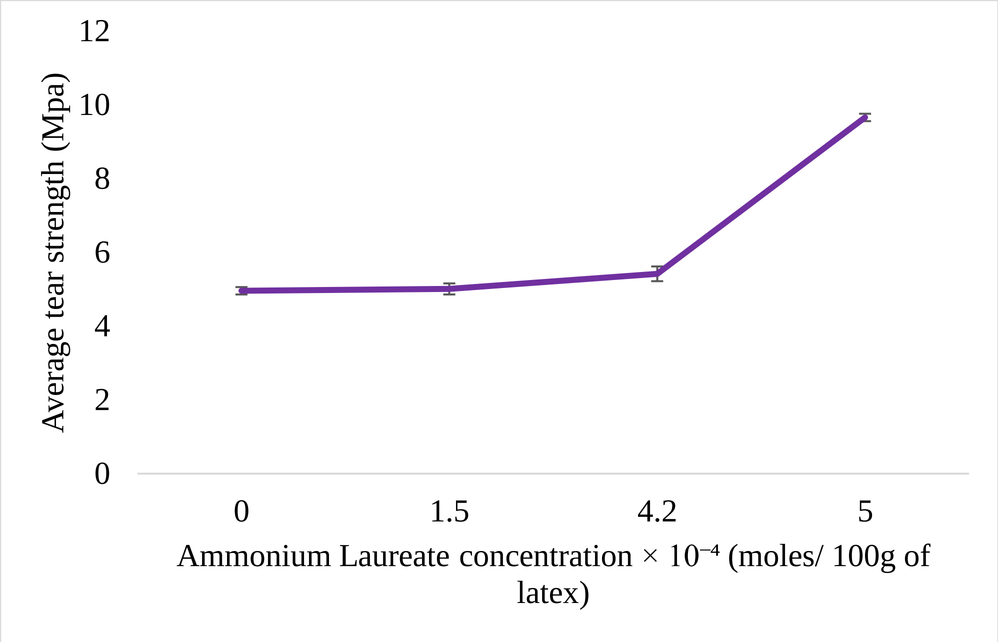

Figure 7: Effect of ammonium laureate concentration on tear strength of latex films

4. Conclusion

Ammonium laurate concentration affects the properties such as KOH number, VFA number, and MST of natural rubber latex. Further, ammonium laurate concentration shows significant effect on properties such as tensile strength, tear strength, and elongation at break of natural rubber latex film. It can be concluded that the ammonium laurate level of 1.63×10^{-4} moles per 100 g of latex will enhance the physico-mechanical properties of natural rubber latex without negatively affecting its chemical properties such as VFA number and KOH number.

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