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To cite this article: M.F. Ab Rahman *et al* 2018 *J. Phys.: Conf. Ser.* **1082** 012011

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The Effect of Alkali Treated Native Sago Starch Concentration on Mechanical Properties of Natural Rubber Latex Composite Films

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Abstract. One of promising methods to improve starch dispersion and/or enhance interfacial strength between starch and rubber matrix is starch treatment using alkali. In this study, the effect of alkali treatment to prepare filler dispersion of native sago starch on mechanical properties of NR latex compounds was investigated. The alkali dispersed native sago starch was prepared with different percentages of total solid content (5, 10, 15 and 20%) at 10% w/w loading of NR latex compounds. Result showed the optimum mechanical properties of alkali dispersed NSS filled NR latex films were achieved at 10% TSC; with 29.74 MPa of tensile strength.

1. Introduction

In recent years, renewable resource fillers such as starch have gained major interest in polymeric composite materials. Starch is a non-toxic biopolymer, inexpensive material, abundant sources, naturally renewable polysaccharide and low environmental impact in which it is readily biodegradable in soil [1 – 2]. Starch has good potential for renewable fillers due to partly crystalline structures, offering interesting properties of polymer composites.

However, a finely dispersed starch in natural rubber (NR) matrix is difficult to achieve due to several limitations of native starch such as hydrophilic characteristic, large particle size and poor process ability [3 – 4]. These limitations lead to the poor mechanical properties of starch/NR latex composite. According to Liu et al. [5], a direct filling of native starch powders causes deteriorated mechanical properties of NR with decreased in tensile strength, tear strength and elongation at break.

Preparation of filler dispersion using alkali is a promising method to improve the distribution of native sago starch (NSS) in a polymer matrix, with proven improved the mechanical properties of NR latex films. A strong alkali is able to disrupt the molecular arrangement within the starch granules resulting from the disruption of intermolecular and intramolecular hydrogen bonding between the water and starch molecules, thus enhancing water solubility of starch [6 – 7].

This study investigates the effect of different total solid contents (TSC) percentages of alkali dispersed NSS (5, 10, 15 and 20%) at 10% w/w filler loading on mechanical properties of NR latex films. Preliminary studies on NSS dispersion have been conducted to characterize the state of dispersion.



2. Experimental

2.1 Materials

NR latex was purchased from Zarm Scientific and Supplies Sdn. Bhd. with the initial properties of dry rubber content (DRC) of 61.10%, TSC of 62.20%, mechanical stability time (MST) of 1213 s, and volatile fatty acid number (VFA no.) of 0.048. Meanwhile, sago starch was obtained from Sago Link Sdn. Bhd. with amylose and amylopectin content ranging from 22 – 31.7% and 68.3 – 78%; respectively. Other compounding ingredients such as diethyldithiocarbamate (ZDEC), zinc oxide (ZnO), potassium oleate, potassium hydroxide (KOH), antioxidant and sulphur were obtained from Bayer (Malaysia) Sdn. Bhd.

2.2 Preparation of alkali-based filler dispersion

The alkali treatment was carried out by mixing NSS with 100g of 5.35% KOH. The mixture was stirred at 200 rpm for 30 minutes before leaving it for two days under room temperature to gelatinise completely. All pH of filler dispersion was in the range of 12.60 – 12.80.

2.3 Preparation of NR latex compound and films

The latex compound was diluted to 50% of TSC with distilled water. The preparation of NR latex compound was carried out in accordance with a previous study [8]. All the ingredients were mixed in the beaker and were constantly stirred for two hours using Wise Stir HS-300 mechanical stirrer at stirring speed of 270 rpm under room temperature. The compound was pre-vulcanized at temperature of 70⁰C in a water bath and until chloroform number 2 (degree of vulcanization of pre-vulcanized NR latex) has achieved. After that, the compound was left overnight under room temperature for maturation purposes. The aluminium plates were dipped into the matured compound and cured in oven for 20 minutes at 100⁰C. The films were stripped by the assistance of calcium carbonate powder.

2.4 Transmission Electron Microscope

The diluted dispersions containing alkali dispersed fillers were deposited onto glow discharged carbon coated grids and allowed to dry. These grids were analyzed using Philips CM 12 CRYO Transmission Electron Microscope (TEM).

2.5 Fourier Transform Infrared Spectrometry

The Fourier Transform Infrared Spectrometry (FTIR) spectrometer used in this study was Perkin Elmer, Model Spectrum One. Analysis was carried out under room temperature within a typical wave number range of 4000 cm⁻¹ to 550 cm⁻¹ under transmission mode.

2.6 Films Mechanical properties test

Tensile test was carried out according to ASTM D412; with the crosshead speed of 500 mm/min by using Instron Machine (Model 3366, USA). The films were cut into dumbbell shape and the recorded results are based on the average of 5 samples.

3. Results and discussion

3.1 Morphological analysis of filler dispersion

Figure 1 (a)-(d) show the states of NSS at different TSC percentages after dispersed in KOH, which were characterized by TEM. Referring to Figure 1 (a), it can be observed that the presence of alkali has destroyed the initial granular form of NSS. The alkali treatment of native starch basically caused starch gelatinization. Gelatinization is the swelling process of starch granule, followed by disruption of granule structures [9]. The distributions of small unreacted core particles which are less than 50 nm were detected in the 5% TSC of alkali dispersed NSS. At 10% TSC of alkali dispersed NSS (Figure 1 (b)), the gel network formation and the increased size of unreacted core particles of the NSS were observed. During alkali treatment, a majority of hydroxyl groups in the anhydrous glucose units are ionized at high pH and therefore, the agglomeration of starch chains and paste retrogradation can be retarded [7].

Starch gelatinization under strong alkali conditions is a heterogeneous fluid-particle reaction in which a liquid (water) reacts with a solid (NSS) to produce a paste, in the presence of an alkali (KOH). Strong alkali weaken molecular network within the starch granules by disrupting hydrogen bonds. This will allow further hydration and irreversible granule swelling which cause an increased in viscosity of the paste [10 – 11]. The starch gelatinization phenomenon was observed by the appearance of gel network, which held together by the formation of local regions of crystallinity that form physical rather than chemical crosslinks.

The ability of strong alkali at low concentration to leach out the amylose in the starch granules particular was reduced by the increase of TSC percentages of NSS where the enhancement of size of unreacted core particles was observed in Figure 1 (c)-(d). The unreacted core particles also agglomerated at 20% TSC of alkali dispersed NSS Figure 1 (d). The appearance of the crystalline region also became less significant which also indicating the increased in amorphous regions in the gel network at high TSC of alkali dispersed NSS. This result also supported by FTIR result (Figure 2).

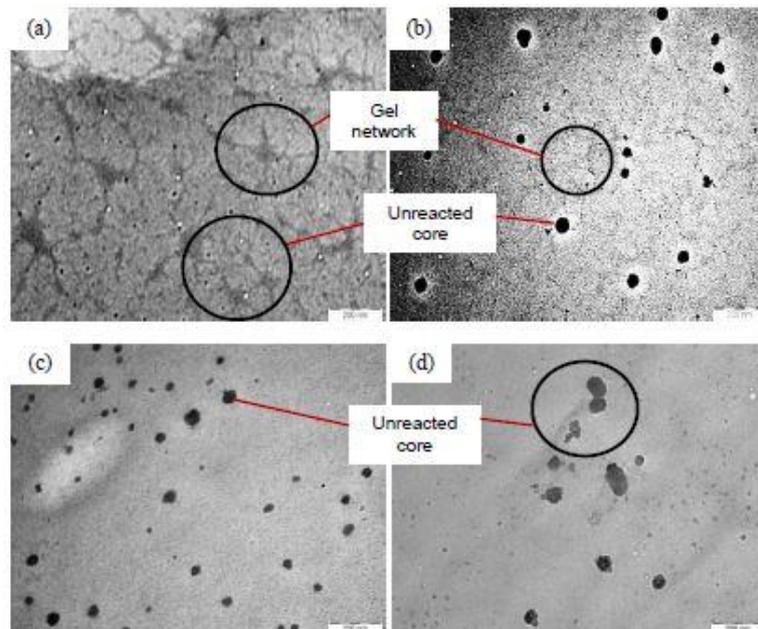


Figure 1. TEM characterization of alkali dispersed NSS at (a) 5% TSC, (b) 10% TSC, (c) 15% TSC and (d) 20% TSC (scale: 200 nm)

3.2 FTIR analysis

The changes in the chemical characteristics of NSS before and after dispersing in alkali medium at different TSC percentages were analysed by FTIR as given in Figure 2. After alkali treatment, all NSS at different TSC percentages showed the transmittance bands became stronger at 3420 – 3250 cm^{-1} and 1640 cm^{-1} due to the increased in O-H groups. As mentioned before, a strong alkali is able to interrupt the molecular arrangement within the starch granules to cause disruption of intermolecular and intramolecular hydrogen bonding between the water and starch molecules, thus enhancing water solubility of starch [7, 12]. The disappearance of bands at 2990 – 2850 cm^{-1} , 1417 cm^{-1} , 1338 cm^{-1} and 998 cm^{-1} were detected which indicated the alteration of CH_2 , C-OH and C-O-C of alkali dispersed NSS. The appearance of a new band at 1020 cm^{-1} was observed and became stronger with the increased in TSC percentages of NSS after alkali treatment. Absorbance at 1020 cm^{-1} is assigned to the vibration of C-O-H deformation and is also related to the amorphous region in gelatinised starch [13 – 15].

Overall, 5% TSC of alkali dispersed NSS exhibited the significant disappearance of the main bands of glycosidic linkages which was indicated the large particle size of starch granules were easily destroyed by alkali at very low starch content. The bands at 1150 cm^{-1} and 1076 cm^{-1} were weakened as well as shifting to 1152 cm^{-1} and 1083 cm^{-1} after alkali treatment. The increased in TSC percentages gave insignificant changes on the bands at 1152 cm^{-1} and 1083 cm^{-1} . The transmittance bands at 1152 cm^{-1} and 1083 cm^{-1} are assigned as C-O, C-C and O-H bonds stretching of C-O-C glycosidic linkages [15– 16]. All filler dispersions for the bands at 860 cm^{-1} , 765 cm^{-1} and 709 cm^{-1} were disappeared and replaced by a broad band at 925-550 cm^{-1} . These results indicate the large particle size of starch granules were destroyed and changed into the gel network of alkali dispersed NSS.

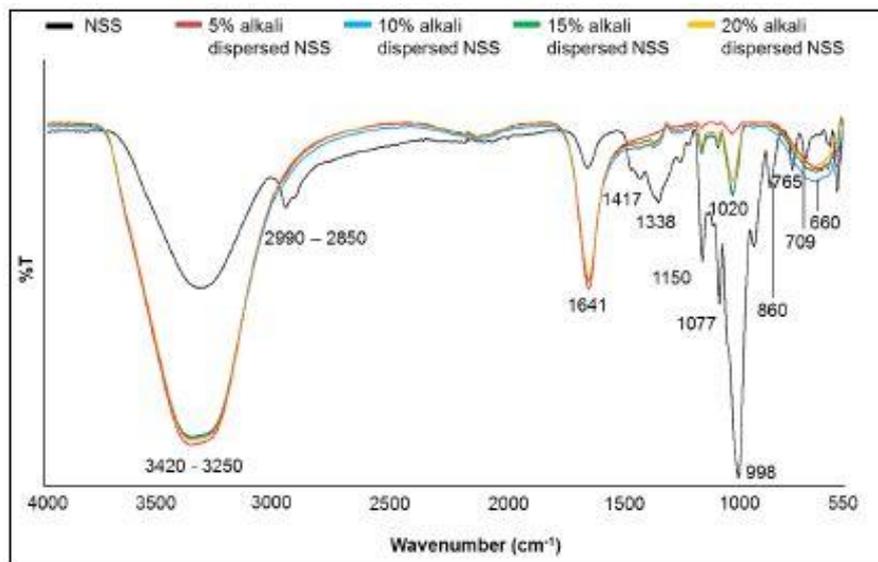


Figure 2. FTIR spectra of alkali dispersed NSS at different TSC percentages

3.3 Mechanical properties of NR latex films

In Figure 3, the stress-strain curves are presented in order to understand the influences of TSC percentages of alkali dispersed NSS on tensile properties of NR latex films. Stress-strain curves show that all curves are divided into three stages. The first stage showed the stress increased gradually at small strains. The second stage exhibited the stress increased at a uniform rate under moderate strains. At the third stage, the stress increases drastically at high strains. This stress behavior attributed to the

strain-induced crystallization; endows these rubbers with good mechanical properties and resistance to crack growth [17].

At first stage, it can be observed that the addition of alkali dispersed NSS at different TSC percentages showed comparable stress of NR latex films before inclined from 150% up to 600% of strain compared to the unfilled NR latex films. This indicates that the addition of alkali dispersed NSS basically increases the modulus or stiffness at higher elongation. The addition of 5% TSC of alkali dispersed NSS lowered the tensile strength as well as elongation at break due to ineffective quantity as filler in NR matrix.

On the other hand, 10% TSC shows significant tensile strength improvement and higher elongation at break values. Further increase in TSC percentage, NSS exhibits significant deterioration in tensile strength due to the poor filler distribution of highly viscous filler dispersions in NR latex matrix.

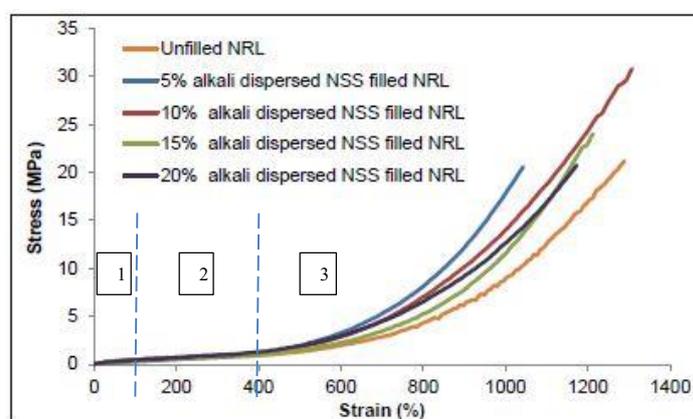


Figure 3. The stress-strain curves of alkali dispersed NSS filled NR latex films at different TSC percentages

4. Conclusion

In conclusion, low TSC of alkali dispersed NSS exhibits good dispersion state, whereas optimum mechanical properties of the NSS/NR latex films can be achieved at 10% of TSC.

Acknowledgements

This work was granted by a FRGS research grant (grant no: 203/PBAHAN/6071353) and laboratory equipment provided by the School of Materials and Mineral Resources Engineering, Universiti Sains Malaysia.

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