Effect of Mercerization and Acetylation on Properties of Coconut Fiber and its Influence on Modified Bitumen

Ivy A. W. Tan, W. H. Wu, Ron A. Chan, and Leonard L. P. Lim

***Abstract*— Coconut fiber, one of many types of natural fibers, is an agricultural waste which is left unutilized after the coconut fruits and juice are extracted. In this research, the effect of the different chemical treatments on the morphological, chemical and physical properties of coconut fiber and its influence on the properties of the modified bitumen were studied. The mercerization effectively altered the surface morphology and reduced the diameter of the coconut fiber. The waxy layer present on the surface of the coconut fiber was significantly reduced after mercerization. Acetylation reported minor reduction on the waxy layer and did not cause any significant changes on the diameter of the coconut fiber. The chemical characterization reported that the hemicelluloses were present only on the surface of the natural coconut fiber whereas the peak of Fourier Transform Infrared spectra associated with the presence of waxes was observed for natural and chemically treated coconut fibers. The bitumen modified with chemically treated coconut fibers exhibited lower penetration values and higher softening point. From the analyses of penetration value, softening point and penetration index, the bitumen modified with 10% NaOH and 50% CH_3COOH treated coconut fibers resulted in enhanced properties for paving binders to be used in warmer region.**

***Keywords*:** Coconut fiber, Mercerization, Acetylation, Modified bitumen

I. Introduction

NOWADAYS, the environment degradation due to fossil based resources is common and inevitable, thus, there is a need to reduce the burden from product that causes adverse effect on the environment and the use of recyclable, biodegradable and sustainable materials such as natural fibers is important to achieve improvement in environmental quality. Bitumen is a complex mixture of organic liquids that is highly viscous, black, and sticky of a predominantly hydrocarbon nature and is the major material used in the construction of road [1]. Bitumen is produced from distillation process of crude oil and only a small fraction comes from natural resources [1]. Bitumen is one of the major materials in asphalt concrete mixtures. Performance of asphalt concrete will deteriorate with respect to time and climate condition; hence by improving the properties of bitumen, it can greatly enhance its performance and lifespan to achieve better pavement performance. Therefore, modification of the bituminous binder is one approach that has been used to improve the performance of asphalt pavement [2]. The utilization of fiber in improving the performance of the asphalt matrix has been recognized from previous researches. It has been reported that fibers can increase the optimum asphalt content in the mixture design and prevent asphalt leakage due to its absorption of asphalts [3]. The use of fiber in the asphalt changes the viscoelasticity, improves moisture susceptibility, creeps compliance and rutting resistance as well as improving low-temperature anti-cracking properties, fatigue life, durability, tensile strength, material toughness and reducing the reflective cracking of asphalt concrete mixtures and pavements [3]. Therefore, based on the literature, it can be deduced that the addition of fiber in asphalt concrete mixture is able to improve its performance. Since most of the previous researches [2-3] were conducted using synthetic fibers that might pose negative environmental impacts, thus, the utilization of coconut fiber which can be obtained from agricultural by-products to improve the properties of bitumen is one of the alternatives to achieve sustainable development. Coconut fiber is one of many types of natural fibers available and it is a renewable and sustainable resource. Besides, coconut fiber is abundantly available in tropical countries such as Malaysia and is amenable to chemical treatment to alter its morphological, chemical and physical properties. This work aims to investigate the effect of mercerization and acetylation treatments on coconut fiber in altering its morphological, chemical and physical properties as well as the influence of the chemically treated coconut fiber on the properties of modified bitumen.

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II. Materials and Methods

2.1. Pre-treatment and chemical treatment of coconut fiber

The coconut fiber was obtained from local market at Kota Samarahan, Sarawak. Prior to chemical treatment, the coconut fiber was cut to 2-5 cm and washed thoroughly with distilled water. Then, the coconut fiber was dried at 60 °C in a convection oven for 24 hours. This was done to remove any impurities or foreign particles present on the surface of the fiber that might affect the treatment process. The pre-treated coconut fiber was subjected to mercerization and acetylation treatments. The concentration used throughout the chemical treatment was in volume fraction (v/v) [4]. Mercerization or alkali treatment of the coconut fiber used sodium hydroxide (NaOH) as the chemical agent. The manipulative variable was the concentration of NaOH which was varied at 10%, 20% and 30% (v/v). The procedure for the mercerization treatment was by soaking the coconut fiber in each of the NaOH solutions with different concentration for 24 hours at 25±1°C. The alkaline treated fiber was then thoroughly rinsed with distilled water to remove the NaOH on the surface of the fiber and the pH was measured to ensure the fiber was neutral (pH 6-7). Then, the NaOH treated coconut fiber was dried in convection oven for 24 hours at 60 °C. Acetylation treatment of the coconut fiber used acetic acid (CH_3COOH) as the chemical agent. Coconut fiber was soaked in 50% CH_3COOH (v/v) at room temperature (25±1°C) for 10 minutes. Then, the acid treated fiber was thoroughly rinsed with distilled water to remove the CH_3COOH on the surface of the fiber and the pH was measured to ensure the fiber was neutral (pH 6-7). Then, the CH_3COOH treated coconut fiber was dried in convection oven for 24 hours at 60 °C.

2.2. Characterization of coconut fiber

Optical microscope with 10x objective and a computer equipped with image analysis and measurement software were used to determine the diameter of the coconut fiber. 15 fibers were tested and the average value was obtained. Image analysis and measurement software V.I.S version 3.00 was used to analyze the fiber images.

Scanning electron microscopy (SEM) was employed to study the surface texture and morphology of coconut fiber and to evaluate the changes on the surface structure provoked by the chemical treatments. The SEM equipment used was. Prior to being analysed by using SEM, the samples were fixed onto an aluminium plate and titanium-coated by using JEOL JFC-1600 auto fine coater to avoid electrostatic charge and to improve image resolution.

Both the natural and chemically treated coconut fibers were examined using Fourier Transform Infrared (FTIR) spectrophotometer to qualitatively identify the surface functional groups of the coconut fiber before and after the chemical treatments. FTIR spectra were obtained after 30 scans and were collected for wavelength ranging from 4000 to 400 cm^{-1}.

Yield test of coconut fiber was carried out on both the natural and treated coconut fiber to determine the changes in the mass of the fiber due to the effect of chemical treatments.

2.3. Bitumen sample preparation

The bitumen was heated with care and stirred frequently to prevent local overheating until it had become sufficiently fluid to pour. Incorporating bubbles were avoided in the sample. Then, the bitumen was poured into the sample container to a depth such that, when cooled to the temperature of the test, the depth of the sample was at least 10 mm greater than the depth to which the needle was expected to penetrate during the penetration test. The standard bitumen sample was prepared by using 80/100 bitumen grade. For the modified bitumen samples, a mass percentage of 1% of the natural and chemically treated coconut fiber was mixed into the bitumen of Penetration-grade 80/100 and the samples were thoroughly stirred during mixing to ensure the coconut fiber was evenly distributed in the bitumen sample. Each container was loosely covered as a protection against dust and allowed to cool to room temperature (25±1°C) for 1 to 1.5 hour.

2.4. Bitumen testing

The penetration test was performed according to the same basic requirements as ASTM D5 or BS 2000 part 49 for penetration of bitumen [5]. This test was used to measure the consistency of the bitumen samples at room temperature (25±1°C). The softening point test of bitumen was performed by using ring and ball method in accordance with ASTM D36 or BS 2000 Part 59 [6]. Penetration Index (PI) is often used to describe the relationship between penetration and softening point [5]. Since asphalt has approximately a penetration of 800 at the softening point temperature, the softening point temperature can be used along with the penetration at 25 °C to determine the temperature susceptibility as:

$$A = \frac{\log(\text{pen at } 25°\text{C}) - \log 800}{(25 - \text{Softening point temperature})} \quad (1)$$

The *A* computed in this manner can then be used to compute PI as follows:

$$PI = \frac{20(1-25A)}{(1+50A)} \quad (2)$$

PI provides an indication of how the hardness of bitumen changes with temperature, specifically its temperature susceptibility [5]. For PI value which is less than -2, it shows that the bitumen has very strong bond to particle; that is Newtonian characteristic. A higher PI with values higher than +2, denotes a lower temperature susceptibility and non-Newtonian characteristic.

III. Results and Discussion

3.1. Morphological characterization analysis

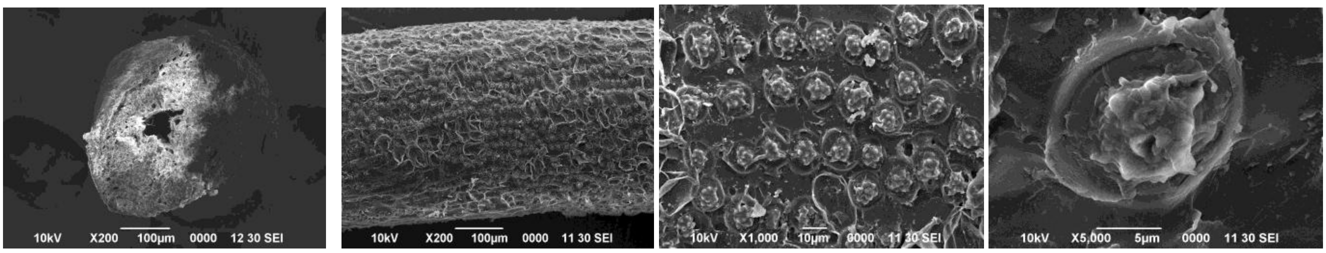
The effect of chemical treatment on the surface morphology and microstructure of the fiber was determined using optical and scanning electron microscopies. Prior to the analysis, it was observed that the colour of the natural coconut fiber was dark brown. The mercerization treatment caused the fiber to exhibit a darker brown colour with the increase in the NaOH concentration whereas the acetylation treatment had slightly lightened the colour of the fiber.

3.1.1. Optical microscopy analysis

From the results obtained from the diameter determination for natural and chemically treated coconut fibers, it was observed that a natural dispersion of values existed for all the fibers studied. The natural coconut fiber presented an average diameter of 221±100 μm, ranging from 90 to 430 μm, showing shape irregularities and large range size. This variation in the diameter of coconut fiber was also observed by Brigida et al. [7], with minimum and maximum values of 69 and 495 μm, respectively with an average diameter of 157±87 μm for the natural coconut fiber. The large dispersion in the diameter of the coconut fiber might be due to the natural variation in the size of the fruit and also the size irregularities of the fiber extracted from the husk of the coconut fruit. Joseph et al. [8] in their studies reported that the average diameter of coconut fiber ranged from 100 to 450 μm. From the literature on coir and other lignocellulosic fibers, the internal structure and properties such as the chemical composition and mechanical properties of lignocellulosic fibers depended on their place of origin, maturity, species, variety, climate, retting degree, decortications and its extraction methods [9-10]. As the coconut fiber in this study was obtained locally, thus, the fiber showed slightly different average diameter and range as compared with coconut fiber obtained from other regions. The treatment with 20% NaOH had caused the highest reduction in the diameter value of the natural coconut fiber and the 20% NaOH treated fiber presented an average diameter of 151±56 μm, ranging from 80 to 270 μm. The results obtained were consistent with the findings reported by Rahman and Khan [11] that alkali treatments such as mercerization would reduce the diameter of fiber, thus, the aspect ratio would increase. The increase in the aspect ratio would lead to the development of a rough surface topography that resulted in better fiber-matrix interface adhesion and an increase in mechanical properties [10]. Besides, in their studies, the highest shrinkage was also observed at 20% NaOH concentration. The reduction of the diameter of the natural coconut fiber might be due to the removal of cementing substances such as lignin and hemicelluloses during the mercerization process.

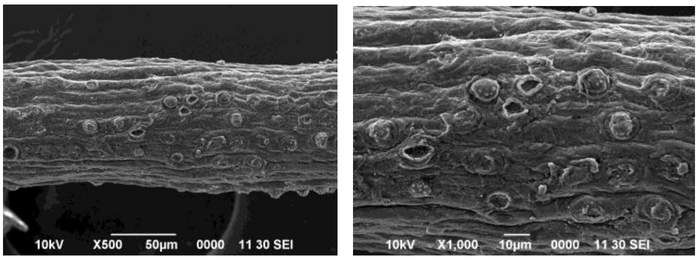
3.1.2. Scanning electron microscopy (SEM) analysis

Figure 1(a) shows the cross-section of the natural coconut fiber which is relatively cylindrical consisting axially oriented cells or fibrils and contains a central hollow part running along the fiber axis which is termed as lacuna. This observation was also reported by Brahmakumar et al. [12]. From Figures 1(b) and 1(c), it could be seen that the surface of the fiber was smooth due to the globular protrusions or patches of waxy layer that were present on the untreated surface of natural coconut fiber at regular intervals. Besides, globular particles, termed as tyloses could be seen to be embedded on the fiber surface which covered the pits on the cell walls (Figure 1(d)). The coconut fibers treated with different concentrations of NaOH exhibited rougher surface, exposing the oriented cells and pits on the cells due to the removal of the waxy layer. Figure 4(d) shows the pit opening after the removal of the tylose from the fiber surface. By comparing the fibers treated with NaOH solution with the natural coconut fiber, it was possible to observe a reduction of the waxy layers. It could also be observed that mercerization process with higher concentration of NaOH showed greater removal of this waxy layer by comparing Figures 2, 3 and 4. This indicated that stronger alkali was more effective in removing the waxy layer from the surface of the coconut fiber. In addition, the surfaces of the mercerized coconut fibers were wavier due to the removal of the parenchyma cells. Similar results were also observed by Brigida et al. [7].



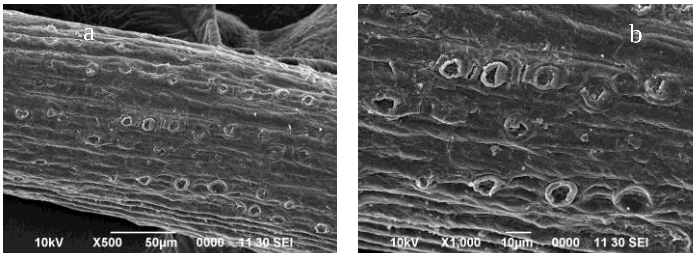
(a) (b) (c) (d)

Figure 1: SEM images of (a) natural coconut fiber cross-section, 200x; (b) untreated fiber, 200x; (c) native surface with waxy layer, 1000x; (d) tylose on the surface of the fiber, 5000x



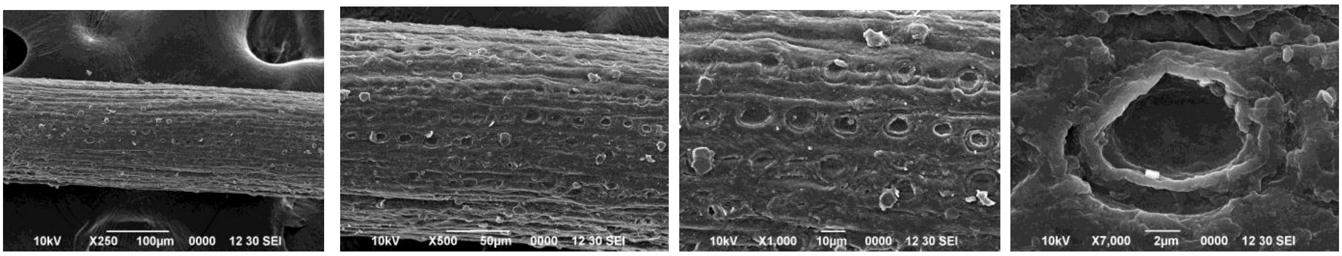
(a) (b)

Figure 2: SEM images of (a) coconut fiber treated with 10% NaOH solution, 500x; (b) native surface of the fiber, 1000x



(a) (b)

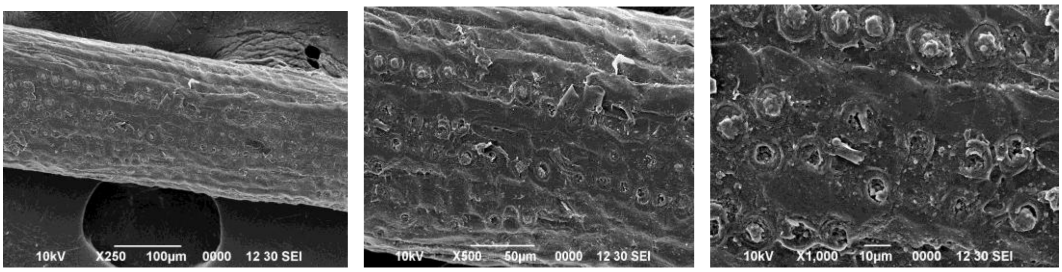
Figure 3: SEM images of (a) coconut fiber treated with 20% NaOH solution, 500x; (b) native surface of the fiber, 1000x



(a) (b) (c) (d)

Figure 4: SEM images of (a) coconut fiber treated with 30% NaOH solution; (b) native surface of the fiber, 500x; (c) native surface of the fiber, 1000x; (d) pit on the surface of the treated fiber after the removal of tylose, 7000x

The acetylation process of coconut fiber by using CH_3COOH (Figure 5) showed that the surface was rougher and wavier as compared to the natural coconut fiber (Figure 1). However, acetylation process did not remove the waxy layer present on the fiber surface as effectively as mercerization process. This might be due to the acid used which was not strong enough and the duration of the treatment was too short to effectively remove the waxy layer from the surface of the fiber. The removal of surface impurities on plant fiber was advantageous for fiber-matrix adhesion since it facilitated both mechanical interlocking and the bonding reaction as a result of the exposure of the hydroxyl groups [11].



(a) (b) (c)

Figure 5: SEM images of (a) coconut fibers treated with 50% CH_3COOH solution; (b) native surface of the fiber, 500x; (c) native surface of the fiber, 1000x

3.2. Fourier transform infrared (FTIR) spectroscopy analysis

The functional groups on the coconut fiber surface for both the natural and chemically treated fibers were characterized using FTIR spectroscopy. The FTIR spectra revealed a strong and broad peak at approximately 3340 cm^{-1} which was assigned to the hydrogen-bonded v(O-H) stretching vibration from cellulose and lignin structure of the fiber. From the spectra, the peak at ~1728 cm^{-1} was observed from the natural coconut fiber only. This peak represented the characteristic bands of hemicelluloses which were present in natural fiber but were not present in NaOH and CH_3COOH treated fibers. Hemicellulose has a random, amorphous structure with low strength which is easily hydrolyzed by dilute acid or base [13], thus the mercerization and acetylation process had reduced, if not removed, the hemicelluloses in the natural coconut fibers. The reduction in hemicellulose due to alkali treatment was also observed by Brigida et al. [7]. The band at ~1240 cm^{-1} was observed in the spectra for all the coconut fibers tested, with the natural coconut fiber showing the most significant peak. This band was associated with the vibration of esters, ethers and phenols groups v(C-O) attributed primarily to the presence of waxes in the epidermal tissue [7]. The significant reduction of the intensity of the peaks for chemically treated coconut fibers in this band was resulted from the reduction of the waxy layer on the treated fiber surfaces due to the chemical treatments. This finding was consistent with the SEM analysis in which the reduction of waxy layer due to the mercerization and acetylation process was observed. All these spectra also revealed a peak at ~1600 cm^{-1}, suggesting the presence of aromatic ring functional group. Besides, the band at 1020 to 1030 cm^{-1} was also observed for all the coconut fibers. This band was assigned to the ring vibration group which indicated the presence of many cyclic compounds. In addition, the peak at ~650 cm^{-1} was observed for all the coconut fibers tested. This peak was in the band position of 600 to 800 cm^{-1} which was assigned to the alkyl halide (C-Cl) functional group. This indicated that some functional groups still remained after the mercerization and acetylation process.

3.3 Yield test analysis

The mercerization and acetylation processes were found to reduce the average weight of the coconut fiber. The mercerization process caused significant weight loss of the coconut fiber in which the weight loss increased as the concentration of NaOH increased. The highest weight loss was recorded at 17.0% which represented the 30% NaOH treated coconut fiber. This might be due to the effective removal of more waxy layers from the surface of the coconut fibers as the concentration of alkali increased. The weight loss of coconut fiber due to mercerization process was also observed by Rahman and Khan [11]. The acetylation process caused lower weight loss in the coconut fiber as compared to the mercerization process. The weight loss recorded was 7.0% for 50% CH_3COOH treated coconut fiber. This lower weight loss might be attributed to the shorter treatment period for the acetylation process in which the waxy layer was not able to be removed effectively as compared to the longer period of the mercerization process.

3.4. Bitumen testing analysis

3.4.1 Penetration value

Penetration test was used to measure the consistency of modified bitumen in order to classify it into specific standard grades. The results obtained for the average penetration value of standard bitumen was 87.6 d-mm. This result was consistent with the grade of standard bitumen used in the testing which was the 80/100 grade bitumen. For bitumen mixed with natural coconut fiber, the average penetration value obtained was 65.1 d-mm and this value was the lowest amongst the modified bitumen. The average penetration values for modified bitumen mixed with 10% NaOH, 20% NaOH, 30% NaOH and 50% CH_3COOH treated coconut fibers were 67.7, 71.3, 74.3 and 65.3 d-mm, respectively. These results showed that the modified bitumen experienced changes in its grade by the addition of coconut fiber. The bitumen grade of the standard and modified bitumen is shown in Table 1. In general, lower bitumen grade was observed for all the modified bitumen, thus, the modified bitumen was harder as compared to the standard bitumen with penetration grade 80/100. Lower bitumen grade is more suitable in warmer regions like Malaysia since lower penetration grades are preferred to avoid softening whereas higher penetration grades such as 180/200 are more commonly used in colder regions to prevent the occurrence of excessive brittleness [14-15].

Table 1: Classification of bitumen grade

Bitumen sample	Bitumen grade
Standard	80/100
Natural coconut fiber	60/70
10% NaOH treated coconut fiber	60/70
20% NaOH treated coconut fiber	70/80
30% NaOH treated coconut fiber	70/80
50% CH_3COOH treated coconut fiber	60/70

3.4.2. Softening point

The softening point obtained for standard bitumen was at 48.7 °C. Since the standard bitumen was of the penetration grade of 80/100, thus, the softening point value obtained was consistent with the requirement of this bitumen grade, as shown in Table 2 [16-17]. For the modified bitumen mixed with natural coconut fiber and 50% CH_3COOH treated coconut fiber, the softening point obtained was identical for both samples which was at 52.0 °C. The modified bitumen mixed with 10%, 20% and 30% NaOH treated coconut fibers showed a gradual increase in the softening point with the increase of the NaOH concentration and the values obtained were 48.3, 58.8 and 62.5 °C, respectively. The modified bitumen mixed with natural, 10% NaOH and 50% CH_3COOH treated coconut fiber were of the penetration grade 60/70, thus, by referring to Table 2, the requirement for their softening point was in the range of 47.0 to 56.0 °C. From the results obtained, the range of the softening point for these modified bitumen samples was from 48.3 to 52.0 °C. Therefore, it could be deduced that the softening points obtained were consistent with the bitumen grade. The modified bitumen mixed with 20% and 30% NaOH treated coconut fiber showed higher increase in the average softening point as compared to the other modified bitumen samples in which the modified bitumen mixed with 30% NaOH treated coconut fiber had the highest softening point. The higher the softening point, the lower the temperature sensitivity, thus, all of the modified bitumen samples, except for the sample mixed with 10% NaOH treated coconut fiber, had shown lower temperature sensitivity as compared to standard bitumen.

Table 2: Requirement for different penetration grade bitumen [16-17]

Bitumen grade	Penetration (d-mm) at 25 °C	Softening point (°C)
Bitumen 60/70	60-70	47-56
Bitumen 80/100	80-100	45-52

3.4.3. Penetration index (PI) analysis

PI is one of the methods for characterizing the temperature susceptibility of bitumen. The PI values obtained for the samples were between -0.050 and +2.626. The modified bitumen mixed with 50% CH_3COOH treated coconut fiber showed the lowest PI of -0.050 whereas the modified bitumen mixed with 30% NaOH treated coconut fiber had the highest PI of 2.626. PI values were generally negative for high temperature susceptibility bitumen and positive for low temperature susceptibility bitumen. According to Roberts et al. [18], PI values between -1 to +1 were mostly good paving binders. Therefore, apart from the standard bitumen sample used in this study, the modified bitumen mixed with natural, 10% NaOH and 50% CH_3COOH treated coconut fibers, with PI values of -0.058, -0.913 and -0.050, respectively were considered as good paving binders.

IV. Conclusions

The mercerization process had significantly reduced the diameter of the natural coconut fiber whereas the acetylation process reported no significant changes on the diameter of the coconut fiber. The mercerization of natural coconut fiber with 20% NaOH concentration reported the highest reduction in its average diameter at 31.4%. The increase in the aspect ratio due to the reduction of the average diameter led to the development of a rough surface topography that resulted in better fiber-matrix interface adhesion and an increase in the mechanical properties of the coconut fiber. SEM characterization showed that the surface of the natural coconut fiber became wavier and rougher due to the removal of the waxy layers and the parenchyma cells from the surface of the fiber by the chemical treatments conducted. The acetylation reported the least reduction in the waxy layers while the mercerization reported higher reduction of waxy layers with the increase of the alkali concentration. From FTIR characterization, the band associated with the vibration v(C-O) attributed primarily to the presence of waxes in the epidermal tissue was observed for all the coconut fibers tested with the natural coconut fiber showed the most significant peak since the chemically treated coconut fiber experienced the reduction of this waxy layer. The weight loss for chemically treated coconut fiber was also reported with the highest weight loss observed in coconut fiber treated with 30% NaOH solution whereas the lowest weight loss was observed for coconut fiber treated with 50% CH_3COOH solution. The penetration values obtained showed that the bitumen modified with the chemically treated coconut fibers exhibited lower penetration values. All the modified bitumen samples showed higher softening point than the standard bitumen except the bitumen modified with 10% NaOH treated coconut fiber. Based on the PI values, the bitumen modified with 10% NaOH and 50% CH_3COOH treated coconut fibers were considered as good paving binders especially for warmer region like Malaysia as compared to the standard bitumen.

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