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Characterization of Mechanical, Physical and Chemical Properties on Thermoplastics Cornstarch Composite Reinforced by Alkaline Treated Coconut Husk Fibre

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ABSTRACT

Natural fibre reinforced composite applications escalates and become so popular recently, this is due to its ability to be a substitute for synthetic based composites. However, the biological element in its structure such as lignin, wax and hemicellulose become a hindrance to the binding factor between matrix and the reinforcement. Many researchers have found ways to overcome this issue, including using chemical treatments such as alkaline treatment. Thus, this paper presents a study on mechanical, physical and chemical properties on thermoplastics cornstarch composite reinforced by alkaline treated coconut husk fibre. The fabricated samples are varied by the wt.% differences between the 10 wt. % NaOH treated coconut husk fibre and thermoplastics cornstarch using the weight ratio of 70%, 50%, 30% and 10%. The mechanical analysis was obtained by the testing of tensile and hardness following the testing standard of ASTM D368 and ASTM D2240 respectively. Additionally, physical analysis was verified by the testing of moisture content following ASTM D 644, density testing following ASTM D 1895 and scanning electron microscope (SEM). Finally, the chemical analysis was determined from the testing of TGA and XRD. Based on the outcomes, in mechanical properties, both tensile and hardness properties of the alkaline treated samples improved due to the great interfacial binding between the coconut husk fibre and the thermoplastic cornstarch. As for physical properties, the SEM shows the binding quality between the reinforcement and the matrix, the alkaline treated sample shown a good adherence between thermoplastic cornstarch and the alkaline treated coconut husk fibre. Furthermore, moisture content and density analyses of the alkaline treated samples have exposed the data of decreasing and increasing trend respectively due to the elimination of element such as hemicellulose, lignin and wax after the coconut husk fibre undergoes the NaOH treatment. On the other hand, the outcomes of alkaline treated samples for chemical analyses displayed the enhancement of crystallinity and a better thermal property of both XRD and TGA. This occurred due to the elimination of amorphous entities and the modification on coconut husk fibre that changed the chemical properties of the alkaline treated bio composites. Overall, this treatment of NaOH has modified the coconut husk fibre and has enhanced the properties of bio composite of thermoplastic cornstarch reinforced with the alkaline treated coconut husk fibre. This research has discovered the detailed comparison between the sample of alkaline treated sample and the untreated sample in mechanical, physical and chemical properties.

Keywords: Bio composite; cornstarch; coconut husk fibre; alkaline treatment; thermoplastic

INTRODUCTION

A composite product is a product that uses a material that consists of two components or more with significantly individual properties that together form a new material that has greater strength, durability, stiffness and wear or fatigue resistance than traditional materials (Ahmed & Chaudhry 2019). Composite materials are usually made with fibre reinforcements, and the most common reinforcement is carbon. These broad properties have led to the discovery of composite materials in mechanical, construction, aerospace, automotive, biomedical, marine and many other industries. The performance of composite materials largely depends on their components and manufacturing techniques (Andrew et al. 2021).

Bio composites are materials made from renewable resources such as plant fibres and natural resources such as wood, paper and metal that are used in a variety of applications such as automotive and aerospace (Akampumuza et al. 2017). Recently, researchers have focused on natural fibres to produce durable products for industry. Many researchers study the mechanical properties of materials made from natural fibres such as kenaf, abaca, grass, hemp, etc (Yıldızhan et al. 2018). Although the strength of natural fibre is lower than that of synthetic fibres, natural fibres are used in the automotive industry and many other products such as ballpoint pen, hook, tennis racket, bicycle, etc (Chakrabarti et al. 2020).

Along the beaches of tropical nations, like Malaysia, coconuts abound. The production of coconut husks and shells has increased due to the growing demand for coconut goods such as coconut water and pulp, which has negative effects on society, the economy, and the environment. Millions of tonnes of coconut husk fibres are produced annually during the processing of the coconut. Malaysia consumes about 745,657.1 million tonnes of coconuts annually (Abdul Wahab et al. 2023).

Studies on the use of coconut husk fibres in composite materials have shown its potential benefits in various applications. Coconut husk fibre, a by-product of coconut processing, has been recognized as a promising reinforcement for thermoplastics and other materials due to its physical, chemical, and surface properties. Additionally, coconut husk fibre as the reinforcement in composite can increase and improve strength, narrow down the environmental issues, and minimize the production cost of composite materials (Danso et al. 2017). Besides, the main advantage is the re-usability of coconut husk fibre can result in reinforced polymer composites that have the desired engineering properties in industry (Cheewawuttipong et al. 2021).

Corn is considered the most important commercially available source of starch in addition to other kind of sources such as wheat, rice, and potato. As a fact, starch is a water-dissolved polymer that has an acceptable mechanical property, and it is a biodegradable polymer (Ibrahim et al. 2019). Other than that, starch is a popular biodegradable material that has many applications due to its abundance, renewability, heat treatment and sufficient thermal stability. Adding cornstarch to plastics to improve their fragmentation and degradation in the environment has attracted great interest. Cornstarch is also widely used as a binder (Sun et al. 2016). Therefore, cornstarch can be one of the reliable matrix components in composite.

Thermoplastics have high performance characteristics and can be mechanically moulded into functional, semi-precision or structural parts that offer a wide range of chemistries, types, properties, and key performance characteristics (Peters et al. 2017). Recently, many researchers focus on biomaterials thermoplastic as a movement towards green industry, thermoplastic starch composites have received considerable attention due to environmental pollution caused by non-biodegradable polymer materials. In addition, Thermoplastic starch compounds are eco-friendly bio-based plastics that can replace petroleum-derived plastic and offer superior properties in packaging applications (Diyana et al. 2021).

In recent years, the application of composite has increased but interest in composites reinforced with natural fibres is growing rapidly due to their many advantages, such as low cost, biodegradability, environmental friendliness, relatively good mechanical properties, and increasing emphasis on the environmental and sustainability (Lotfi et al. 2019). However, one of the disadvantages of bio-fibres is, it contains other extractives substances such as wax, pectin, and other water-soluble compounds. Other than that, natural fibre is poor adhesion between fibre and matrix, it also contains cellulose, moisture absorption and vacant at the fibre-matrix bonding surface, which cause dimensional errors that affect the engineering properties (Aravindh et al. 2022).

Many processing methods have been developed to overcome the natural problems of natural fibres. Chemical treatment is one of the ways to enhance natural fibres properties especially on surface geometry, removing dirt, the strength of fibre, and fibre-matrix bonding factors. A chemical treatment often used in natural fibres is an alkaline treatment. Alkaline treatment is expected to remove large amount of the hemicellulose, waxes, lignin, and alkali-soluble elements, resulting in roughening of the fibre surface due to reduced fibre aggregation (Arsyad et al. 2015).

Coconut and coconut husk fibres reinforced polymer composites have improved stiffness, flexural strength, and hardness with longer fibre length, while impact energy reduction and water absorption properties remain unchanged (Obada et al. 2020). However, coconut husk fibres-based composite has several problems, one of the problems is the mechanics of fibre reinforcement is coconut husk fibre hydrophilicity and the existing of the extractives elements that become hindrances to the binding effects. Due to this hydrophilicity, the proper interface strength is not achieved, and it negatively affected the mechanical properties of the composite material (Verma et al. 2023).

There is a lot of researchers make a study that has a same interest of chemical treatment on coconut husk fibre, the coconut husk fibres were immersed in 5% NaOH solution at laboratory temperature for various period, rinsed and dried. SEM (scanning electron microscope) analysis showed that alkaline treatment in NaOH solution led to gradual removal of the binder from the coconut husk fibre surface and makes it rougher. The effect of alkali changes on the surface of the fibres and increases the percentages of binding abilities between the fibres and the matrix (Valášek et al. 2021).

In conclusion, this research is aiming to evaluate the effects of alkaline treatment on coconut fibre to improve the properties of thermoplastic composite, reinforce between treated coconut husk fibre and cornstarch. This research will contribute the improvement in chemical, mechanical, physical, and environmental properties. Otherwise, this research aims to reduce the problem of coconut husk fibre waste and our dependence on non-biodegradable materials so that we can move towards a more sustainable and environmentally friendly future.

This research is aiming to evaluate whether the treated fibre composite is more convenient or better than the normal or untreated fibre composite in terms of chemical composition, bonding between matrix and reinforcement and mechanical properties. Specifically, the objectives are to fabricate thermoplastic cornstarch composite, reinforce with untreated coconut husk fibre and thermoplastic cornstarch composite reinforce with alkaline treated coconut husk fibre. Other than that, this investigation also aims to determine mechanical and physical properties between thermoplastic cornstarch composite reinforce with untreated coconut husk fibre and alkaline treated coconut husk fibre. Besides, this research is to evaluate analysis of chemical properties between thermoplastic cornstarch composite reinforce with untreated coconut husk fibre and alkaline treated coconut husk fibre. Following that, this study is filling the research gap about effect of alkaline treatment on coconut husk fibre on the aspect of crystallinity and thermal stability resulting the improvement of the bio composite durability and the enhancement of the mechanical and physical properties of the bio composite.

METHODOLOGY

MATERIAL

The coconut husk fibre or coconut peat was purchased from the Farmer Boy Company located at Johor Bahru. The coconut husk fibre was in fined, powdered form. Additionally, the coconut husk fibre is 100% pure without any foreign and unnecessary composition. The small amount of sieved coconut husk fibre for untreated and treated sample has been collected for particle size analysis (PSA). The result of particle size analysis showed that, the range of the coconut husk fibre size (PSA) is between 34.674µm until 549.541µm. The cornstarch is provided by Polyscientific Enterprise Sdn. Bhd. Glycerol, it is an odourless, colourless, a high viscosity liquid, and non-toxic. In this study, the glycerol was selected as the plasticizer to produce thermoplastic cornstarch, it was purchased from QReC (Asia) Sdn Bhd. The pellet form of NaOH from EMD Millipore Corporation is chosen for this investigation.

FIGURE 1. Coconut husk fibre.

FIGURE 2. Cornstarch in powdered form.

ALKALINE TREATMENT ON COCONUT HUSK FIBRE

The alkaline treatment methodology on coconut fibre is referred to a study by Seok et al. (2019). For the alkaline treatment, NaOH 10wv. % solution was prepared by dissolving 10g NaOH into 1000ml distilled water. After that, to expel contaminant, the coconut husk fibre has been washed with distilled water. Subsequently, the clean coconut husk fibre has been air dried in room temperature for 24 hours. Following that, 100g coconut husk fibre has been measured, and it has been soaked into 1000ml NaOH at room temperature for 24 hours. After a while, the treated coconut husk fibre was washed with distilled water to eliminate the excess NaOH. Then, the coconut husk fibre was once again has been air dried for 24 hours. Finally, the coconut husk fibre has been dried in the oven for 8 hours at 60 degrees Celsius.

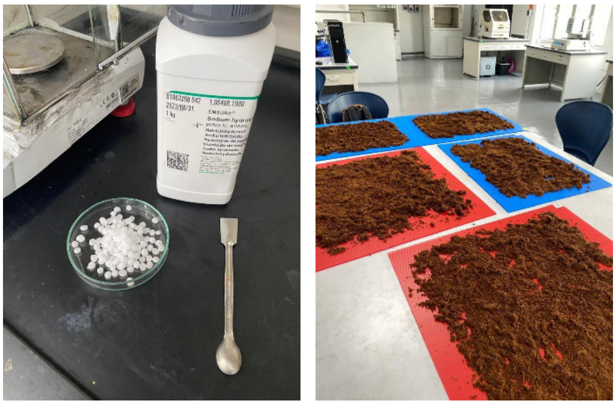

FIGURE 3. Process of alkaline treatment on coconut husk fibre.

SAMPLE PREPARATION

The methodology of making the sample of thermoplastic cornstarch mixing involved two main materials: the cornstarch and glycerol. Using the ratio of 70% of cornstarch and 30% of glycerol from the desired for producing thermoplastic cornstarch mixture. Following that, the materials has been stir until it well distributed. Once the mixed cornstarch and glycerol is prepared, by weighing alkaline treated coconut husk fibre constituting 70% of the total required weight, combined it with 30% mixture of cornstarch and glycerol. Then, stir it until it is well mixed. The process remains identical, but with different ratios of alkaline treated coconut husk fibre to the thermoplastic cornstarch by 50%, 30%, 10%, and 0%. The Go Tech motorise hydraulic moulding test press machine has been heated until the temperature of the press mould reaches 165 ^{o}C. The machines pressure is manually set until it reaches a constant pressure of 50 kg/cm2. Each sample fabrication is consistent which all the parameter of time, pressure and temperature are standardized and carefully maintained.

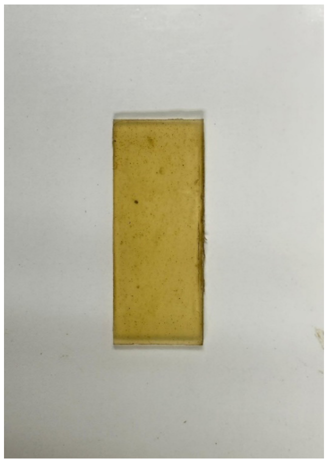

FIGURE 4. Sample of thermoplastic cornstarch

TENSILE TESTING

Tensile testing was selected to be one of the tests to analyse the sample's mechanical properties, characteristics, and behaviours. In this test, ASTM D368 is the testing standard that is chosen to perform the tensile test. Following that, Shimadzu Autograph AGS-X Series, and the crosshead speed of 2mm/min were applied. Accordingly, 5 samples of each various ratios of thermoplastics cornstarch reinforced with alkaline treated coconut husk fibre were utilized in this test, whereas the samples were prepared with the required dimensions of length, width, and thickness by 140 mm x 13 mm x 3 mm respectively.

HARDNESS TESTING

After tensile test, the second mechanical testing that has been chose is Shore A Hardness Testing. To perform the test, ASTM D2240, the standard testing was utilized in this study. Following that, Teclock Durometer was used. Importantly to mentioned, the test utilized 5 readings of each various ratios of thermoplastics cornstarch reinforced with both untreated and alkaline treated coconut husk fibre, whereas the samples were prepared with the required dimensions of length, width, and thickness by 140 mm x 15 mm x 3 mm respectively.

SCANNING ELECTRON MICROSCOPE (SEM)

The morphological characteristics of samples were obtained using a scanning electron microscope (SEM) following the practices standard of ASTM E986. The Zeiss Evo 18 Research model from Jena, Germany was utilized in this physical analysis, whereas the machine operated at acceleration voltage of 10 kV. The samples were taken from tensile test, following that, the samples were prepared in required sizes. The sample's surface was coated with a layer of gold before conducting the SEM analysis.

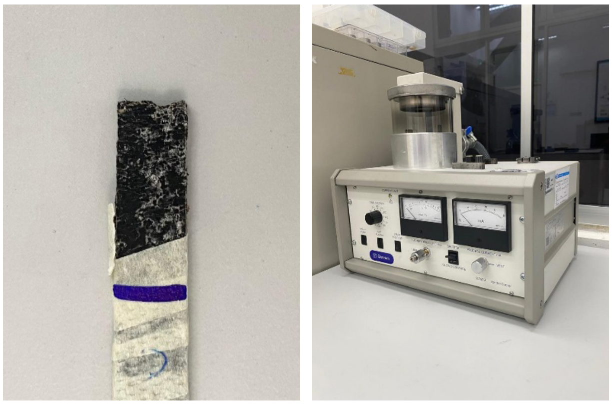

FIGURE 5. The post-tensile sample has been used for SEM analysis.

MOISTURE CONTENT TESTING

The sample's moisture content value was obtained by conducting moisture content test whereas the test was followed ASTM D 644 as a testing standard. To determine the measurement of the initial weight, W_i and the final weight W_f of the sample, weighing balance was used while conducting this test. In this test, 3 samples of each various ratio were utilized. Next, each of the sample was cut with the dimension of 10mm (length) x 10mm (width) x 3mm (thickness), then, the sample has been weighted to get its initial weight, W_i. Following that, as for drying process, the sample has been oven dried for 24 hours at 105°C ± 2°C. Subsequently, right after the sample were removed from the oven, the final weight of the sample, W_f, is measured immediately to avoid any surrounding factor affects the final weight. The moisture content for each sample was obtained by calculated the recorded data of initial weight and final weight by using the equation of moisture content as shown in equation (1).

$$Moisture\ content, MC\ \% = \frac{(w_f - w_i)}{w_i}\ X\ 100\% \qquad (1)$$

FIGURE 6. Moisture content procedure of weighting before oven dry and weighting after the oven dry.

DENSITY TESTING

The testing of density was following the standard of the ASTM D 1895. First, the sample of 10mm (length) x 10mm (width) x 3mm (thickness) in different ratios are prepared. Then, the samples undergo the drying process in an oven for 24 hours with a constant temperature of 105°C ± 2°C to eliminate the existing moisture content in the sample. In this analysis, the experiment was conducted by using electronic densimeter. Following the drying process, the sample was weighted to get the dry mass of the sample. After that, the sample has been submerged into the water part of the densimeter to obtain the density and the volume. The density value was determined from using the electronic densimeter. The formula of the density is written in equation (2)

$$Density = mass/volume \qquad (2)$$

X-RAY DIFFRACTION ANALYSIS (XRD)

The untreated and treated samples of thermoplastic cornstarch and coconut husk fibre were cut into small pieces X-ray diffraction (XRD) has been conducted to determine the information about the crystallographic structure and chemical composition of the samples. In this testing, the thermoplastics cornstarch reinforced with untreated coconut husk fibre and thermoplastic cornstarch reinforced with alkaline treated coconut husk were used as the sample throughout this testing. Additionally, Rigaku Miniflex spectroscopy ray diffraction spectroscopy ray diffraction instrument has been utilized for this testing. Following that, the X-ray instrument radiation beam was using Copper Kα (λ = 0.1540 nm), additionally, other operating conditions were set by 30 mA, 40 kV, and a two-θ range of 5–50 C. This method is utilizing an equation whereas the equations equate between intensity of both crystalline and the amorphous. The equation (3) is used based on Segal method (Nam et al. 2024).

$$CI\,(\%) = (I200 - Iam)\ /\ I200 \times 100\% \qquad (3)$$

THERMOGRAVIMETRIC ANALYSIS (TGA)

The untreated and treated samples of thermoplastic cornstarch and coconut husk fibre were cut into small pieces weighted between 5-15mg. The analyses were made by using Tg 209 F3 Tarsus by Netzsch for analysing the thermal properties for untreated and treated samples. The parameter that has been set for the analysis are: 50 mL/min flow rate of dynamic nitrogen atmosphere surrounding, the range of temperature between 25 to 600 °C, with a constant heating rate of 10 °C/min−1. The result is obtained from TGA graphs of the percentage of weight loss versus temperature to analyse the thermal properties of the samples (Kamaruddin et al. 2022).

STATISTICAL METHOD OF ANOVA

The statistical method that has been utilized in this research are the One-way Analysis of Variance (ANOVA) whereas the ANOVA is used for assessing the significance data of each group by using Duncan's multiple range of $P < 0.05$.

RESULTS AND DISCUSSION

TENSILE ANALYSIS

Figure 7 displays the graph for tensile strength of thermoplastic cornstarch reinforced with untreated coconut husk fibre and alkaline treated coconut husk whereas it consequently has various fibre content percentage as the control factor.

From the graph, it illustrates that the increments of coconut husk fibre loading increased the tensile strength and tensile modulus for both untreated and alkaline treated fibre. In details, the tensile strength at 0% of fibre loading is the lowest with the value of 0.76 MPa while 70% is the highest, both values are at 25.15 MPa for untreated samples and 27.99 MPa for treated samples respectively. From this, the fibre loading plays a good role for improving the mechanical properties. This finding has been mentioned in Mogan et al. (2023) whereas the research has concluded that the amount of fibre in a bio composite is crucial where it comes to tensile properties. In the graph, the small standard deviation bar shows at sample of 0%, 10% illustrates the data are closer from the mean while the other samples have wider standard deviation bar indicates the data are more variable from the mean.

Furthermore, the composite that reinforced with alkaline treated coconut husk fibre has improved tensile properties compare to the composite with untreated coconut husk fibre reinforcement. The tensile strength of this composite with alkaline treated fibre sample was increased compared to untreated bio. In details, the improvement between the untreated and treated bio composite are significant. At 10% fibre loading, the tensile strength improved the most by 104.89%. Following that, the least improvement in tensile properties for alkaline treated fibre is at 30% whereas it was increased by only 8.32% compare with untreated fibre. At 50% the improvement is about 13.21%. Finally, at 70% fibre loading, the improvement percentage was approximately 11.31%.

Related to this, there are several factors might be affected the improvement in tensile properties of the composite with alkaline treated coconut husk fibre. Generally, the first factor is a major elimination of the unwanted elements on the fibre such hemicelluloses and lignin whereas it revealed the fibre groove and rough surface. This finding is related to what written in Kamaruddin et al. (2022), it also said the removal of those unwanted elements helps the rearrangement within the direction of the tensile deformation. In additional, it also enhances the properties of tensile. Following that, the alkaline treatment resulting a great interfacial binding between the matrix and the reinforcement due to the fibre's rough surface.

Referring to Yik Ru Ng et al. (2018), alkaline treatment can minimize the unwanted elements on the fibre's surface which led to the improvement of the physical properties of the combination between matrix and the reinforcement. Besides that, alkaline treatment also removes the hydrophilic components in fibre, resulting and enhancement of the tensile properties of the composite.

Table 1 below shows the tensile properties analysis of variance result for both treated and untreated sample. Additionally, the result of ANOVA shows the P-value for both of untreated and treated sample were less than 0.05. This indicates that there are significant variations in the mean of tensile strength between the various ratios of the composite levels.

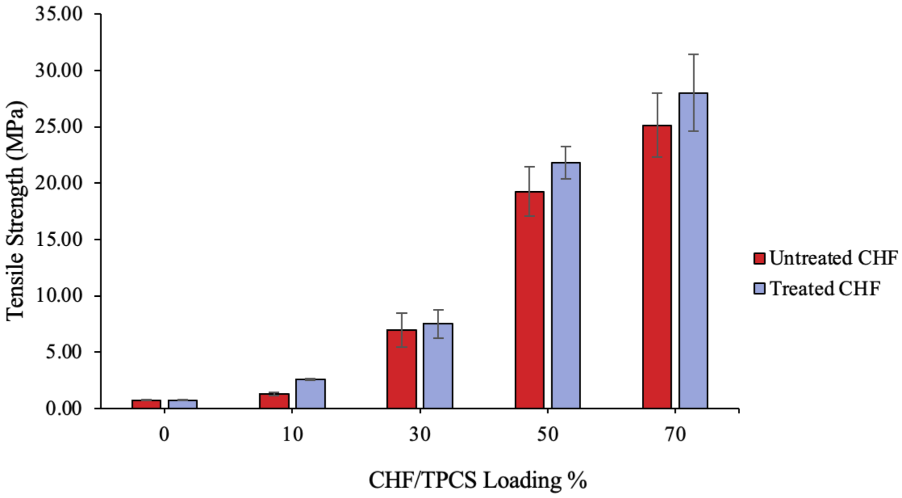

FIGURE 7. The tensile strength value of the untreated and alkaline treated samples.

TABLE 1. The tensile strength analysis of variance.

Type of sample	df	P-value Tensile Strength
Untreated sample	4	2.20728×10^{-08}
Treated sample	4	8.82706×10^{-09}

HARDNESS ANALYSIS

Figure 8 below exposes the hardness value of two types of samples such as thermoplastic cornstarch reinforced untreated coconut husk fibre and thermoplastic cornstarch reinforced with alkaline treated coconut husk fibre.

The graph above illustrates there is an increment hardness value as the coconut husk fibre loading content rises. The pure thermoplastic coconut husk fibre is taken as the control samples, has unveils the lowest value of hardness between the samples by the value of 25.77. Consequently, at 10% of fibre loading for both untreated sample and the alkaline treated samples, the hardness value continues to increase with the value of 45.13 and 52.27 respectively. Furthermore, the hardness value of 30% and 50% for both untreated samples and alkaline treated samples are constantly inclined. Following that, the highest hardness value resulted at the fibre content of 70% for both untreated and alkaline treated samples with the value of 80.9 and 88.4 respectively. In the graph, the small standard deviation bar at treated sample of 10%, 50% and 70% shows the data are closer from the mean while other samples have wider standard deviation bar indicates the data are more variable from the mean.

The outcomes that have been achieved are attributed due to few scientific factors. According to a study by Abbass et al. (2020), it discovered that the increment of coconut husk fibre content in the bio composites led to reduction in matrix thickness. This factor is one of the causes that make the bio composites can resist the penetration and has high hardness values. Meanwhile, the other factors that might be related to the improvement of particle rigidity and that has led to the improvement of the bio composites' stiffness. This statement can be validated by research that has been made by Homkhiew et al. (2024).

Other than that, the bonding between the higher loading of coconut husk fibre and the thermoplastic cornstarch becomes harder due to the movement hindering in the stress direction which it has caused the enhancement of the hardness properties of the bio composite. This is also has been quoted in a study published by Njom et al. (2022).

On the other side, another factor that also attributed to this study's outcome is the alkaline modification of the coconut husk fibre. The trend proved that the alkaline treatment has enhanced the hardness properties of the bio composites as mentioned by Raja et al. (2021). In addition, this phenomenon is also related to a study that was conducted by (Sahu et al. (2020) whereas it states that the surface of the coconut husk fibre has been altered during alkaline treatment. Other that than, this treatment also improved the matrix-fibre adhesion which led to the enhancement of the bio composite compatibility and strengthen the bio composite. Correlated to this finding, Jahan et al. (2020) investigated and stated that the coconut husk fibre that undergoes the alkaline treatment has improved the packing factors that also resulted to low number of void formations. Therefore, this characteristic makes the medium of the bio composite to have a good and effective pressure transfer which can prove the improvement of the indentation resistance of the bio composite.

Table 2 below shows the hardness properties analysis of variance result for both treated and untreated sample. Additionally, the result of ANOVA shows the P-value for both of untreated and treated sample were less than 0.05. This indicates that there are significant variations in the mean of tensile strength between the various ratios of the composite levels.

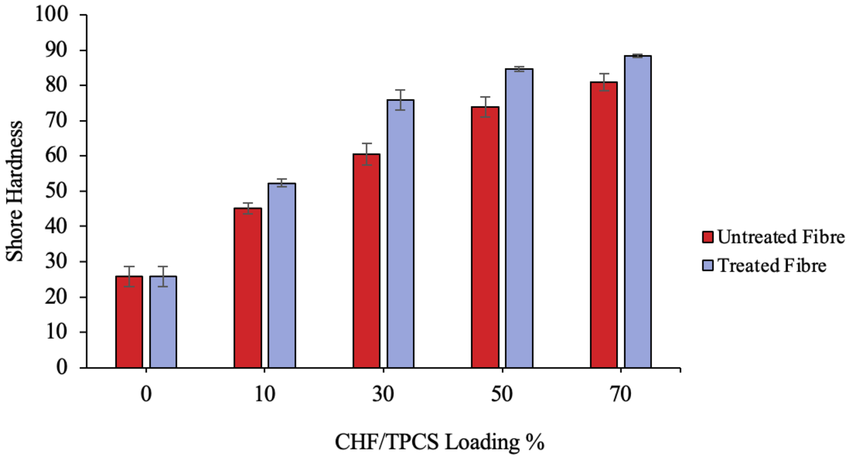

FIGURE 8. The hardness value of the untreated and alkaline treated samples.

TABLE 2. The hardness analysis of variance.

Type of sample	df	P-value Hardness
Untreated sample	4	9.57484×10^{-10}
Treated sample	4	1.01788×10^{-11}

SCANNING ELECTRON MICROSCOPE (SEM) ANALYSIS

Figure 9 portray the untreated coconut husk fibre and treated coconut husk fibre at 50µm. The analysis revealed the differences of the structure of both fibres. The untreated coconut husk fibre has smooth surface whereas it indicates the surface of the untreated coconut husk fibre is like to be wrapped by the smooth elements such as wax, pectin and impurities. The presence of the substances that wrapped the fibre enclosed the bulges and grooves that naturally existed on coconut husk fibre. On the other hand, the alkaline treated coconut husk fibre has rough surface showing that the NaOH has eliminated some lignin, pectin, hemicellulose and other materials in coir fibre. The absence of these elements exposed some bulges and grooves on the coconut husk fibre. This finding can be agreed by Barrera-Fajardo et al. (2024), in the journal said that the coconut husk fibre naturally have a smooth surface due to the present of non-cellulosic elements such as wax layer that enclosed the fibre's surface. In addition, when the NaOH solution is merged the coconut husk fibre, it reacted with the hydroxyl groups that naturally existed on fibre, this reaction breaks up the elements of lignin, hemicellulose, and other unwanted impurities. Following that, this treatment resulting a surface that naturally smooth has improved the surface roughness on the fibre.

The improvement is taking an important role due to the reason that it could enhance the mechanical binding of the fibre in the matrix. This interlink creates a strong physical interface connection between the coconut husk fibre and thermoplastic cornstarch. Therefore, this morphological characteristic that has enhanced related with the structure's hierarchy of the fibres whereas it may influence their properties especially in mechanical properties (Valášek et al. 2021).

Figure 10 above is a result of pure thermoplastic cornstarch under SEM at 100µm. The structure of the thermoplastic cornstarch displays a completed 'cooked' whereas there is no powder or uncooked starch embedded in the pure thermoplastic cornstarch. Related to the result of tensile strength that shows the improvement in mechanical properties after alkali treatment on the coconut husk fibre proved that this treatment helps the mechanical bonds between the matrix and the reinforcement of alkaline treated coconut husk that also written by Mahmud et al. (2023), that said the alkaline treatment enhanced the crystallinity of the fibre and it also improved the attachment of matrix on fibres.

Figure 11 (left) illustrates a poor bonding between thermoplastic cornstarch as the matrix and the untreated coconut husk fibre as the reinforcement. In additional, there is gap between the fibre and the matrix. This characteristic may contribute to poor mechanical performance of the composite. Compared to right figure, SEM shows a good adherence between thermoplastic cornstarch and the alkaline treated coconut husk fibre, and the bonding is completed. Both are at 100µm.

A study by Shaofeng Ru et al. (2022) also mentioned the alkali treatment enhancing interfacial bonding between the fibre and the matrix. By mechanism of the composites, the melted matrix when heated can flow or enter the bumpy and the groove of the fibre's surface creating a good and strong mechanical interlocking between the composite elements of matrix and the reinforcement and get even stronger after solidification phase.

Other than that, the composite sample also contained few discontinuities that might affect the mechanical performance of the bio composite. In Figure 12 portrays that SEM has detected voids and pores at 100µm. This might be occurred due to moisture vaporization during hot press.

Besides that, the crack propagation occurred due to the existed discontinuities or because of the fibre orientation that is not properly oriented in the matrix. These factors need to be eliminated to improve the mechanical performances of the composites.

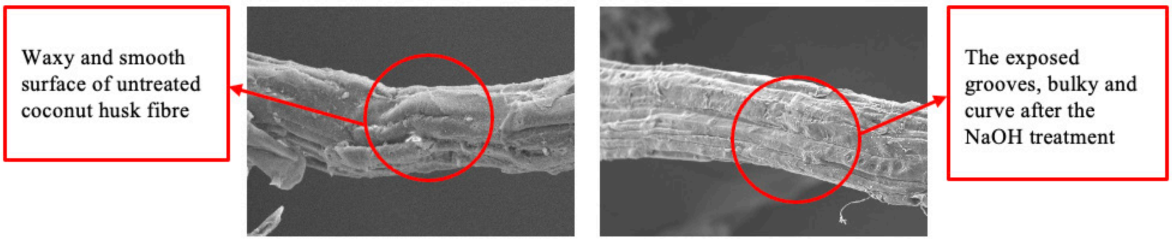

FIGURE 9. The untreated coconut husk fibre and treated coconut husk fibre under SEM at 50µm.

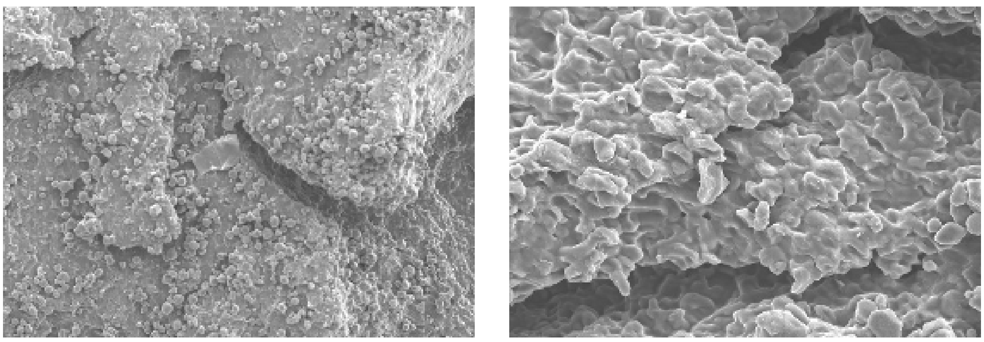

FIGURE 10. The pure thermoplastic cornstarch under SEM at 100µm.

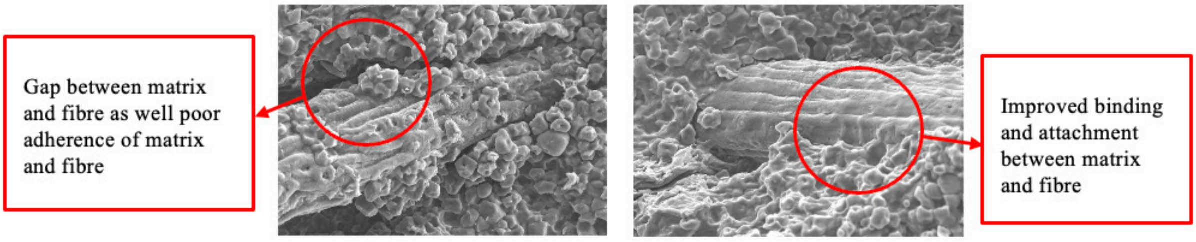

FIGURE 11. The untreated sample (left) and treated sample (right) under SEM at 100µm.

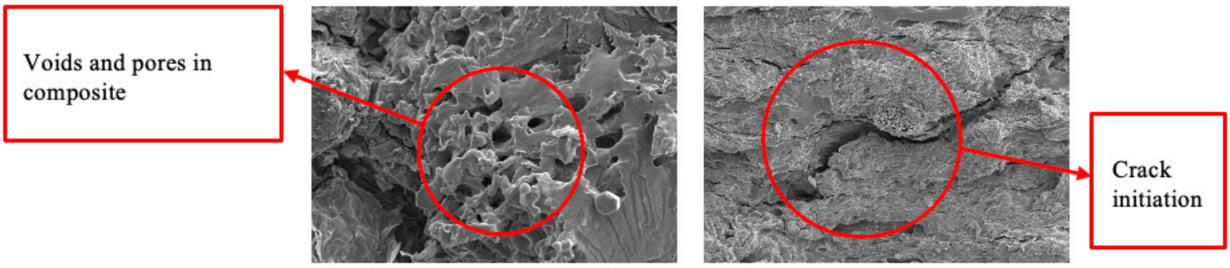

FIGURE 12. The voids, pores and crack initiation in sample under SEM at 100µm.

MOISTURE CONTENT ANALYSIS

Figure 13 shows the graph of moisture content between thermoplastic cornstarch reinforced with untreated coconut husk fibre and alkaline treated coconut husk whereas it consequently has various fibre content percentage as the manipulated factor.

The graph illustrates there is a decrement of moisture content in every increment of fibre loading content for both untreated and alkaline treated coconut husk fibre. Additionally, the highest moisture content is at pure thermoplastic cornstarch with the value of 14.24 %, this property is taken as the control sample. Moreover, as the loading of the coconut husk fibre increased, the moisture dropped accordingly, whereas the lowest moisture content is at 70% of fibre loading content at the value of 4.24 % for untreated sample and 3.6% for treated samples. A study conducted by Mogan et al. (2023) also found that the moisture content decreases as the fibre loading percentage increases. In the graph, the small standard deviation bar for treated sample with the fibre loading of 0%, 10% and 70% shows the data are closer from the mean while the rest samples show wider standard deviation bar indicates the data are more variable from the mean.

Furthermore, in the graph, the treated samples demonstrate an orderly trend of lower percentage of moisture content. In details, at 70% of fibre loading, the moisture content of the alkaline treated samples is 15.09% lower that the untreated samples. While at 50% and 30 % of fibre loading, the moisture content for alkaline treated sample is also lower than the untreated sample by 20.55% and 27.7% respectively. At 10 % of fibre loading the moisture content differences between the alkaline treated sample and untreated sample is 9.59%.

This finding found that the untreated sample that contained highest percentage of moisture might be due to the hydrophilicity of pure coconut husk fibres whereas in the structure of pure coconut husk fibre contains hydroxyl group. Other than that, this investigation also showed a significant moisture content dropped in the alkaline treated bio composites. This data proved that the alkaline treatment improved the interfacial binding between the thermoplastic cornstarch and treated coconut husk fibre which this property has minimizing water or moisture from entering the interface. Besides, this treatment of NaOH allowed the matrix and reinforcement to bind effectively. This finding corresponded with the discoveries of past studies by Kamaruddin et al. (2022).

Similarly, a study that has been conducted by Sari et al. (2017) mentioned that the higher number of moisture contents for the untreated sample can affect the mechanical properties due to its microfibril degradation and decompose occurred. Comparatively, the alkaline treatment on coconut husk fibre resulting low percentage of moisture content. This result indicate that the alkaline treatment has eliminated the hemicellulose content in the fibre. The alkaline treatment also reduces the water accessibility. The minimum number of moisture content is relatively improved the properties of the bio composite. Therefore, the alkaline treated coconut husk fibre has enhanced the bio composite.

Table 3 below shows the moisture content analysis of variance result for both treated and untreated sample. Additionally, the result of ANOVA shows the P-value for both of untreated and treated sample were lower than 0.05. This indicates that there are significant variations in the mean of moisture content between the various ratios of the bio composite.

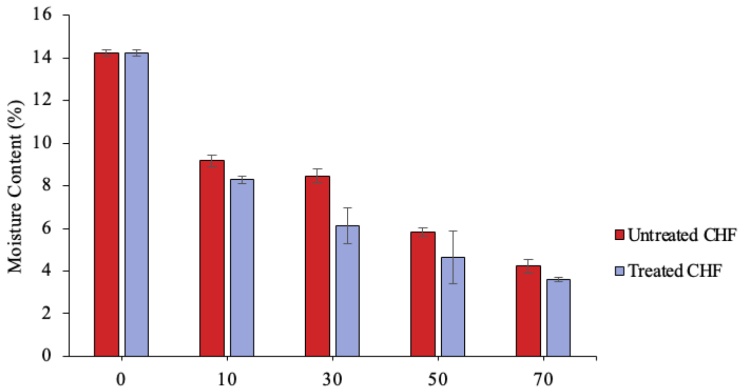

FIGURE 13. The moisture content percentage of the untreated and alkaline treated samples.

TABLE 3. The moisture content Analysis of Variance.

Type of sample	df	P-value Moisture Content
Untreated sample	4	$6.47796x^{-12}$
Treated sample	4	$2.57159x10^{-08}$

DENSITY ANALYSIS

Figure 14 below illustrated a density graph comparison between the alkaline treated and untreated coconut husk fibre in thermoplastic cornstarch composite whereas it consequently has various fibre content percentage as the influenced variable.

In general, the percentage of density inclines as the fibre content loading raises. The trend illustrates that the density at 0% of fibre content is the lowest at the value of 1.415 g/cm3 while the highest density percentages is at 70% of fibre content with the value of 1.451 g/cm3 for untreated sample and 1.482 g/cm3 for alkaline treated sample. Following with that, the density value at 10% of fibre content for alkaline treated sample is slightly lower than the untreated sample by 0.42%. In addition, the density of the alkaline treated sample displays more noticeable increase especially at the fibre content of 50% and 70% compared to the untreated sample. This trend is correlated with a study by Mogan et al. (2023). In the graph, the sample of 0%, 1-%, 30% and 50% of untreated samples have small standard deviation bar shows the data are closer from the mean while the others have wider standard deviation bar indicates the data are more variable from the mean.

From the finding, the investigation has shown that the increment of fibre content has raised the density percentage. A study that been conducted by S. Mahmud et al. (2021) found that the increment of the fibre has also raised the void content in the bio composites that automatically increase the density number. Plus, it proved that the rise of void development is attributed by the increment of fibre content, this reason also been stated in a study by Das et al. (2016). Additionally, the density could also be affected by the porosity of the bio composites. This finding also stated that the increment of density improved the mechanical properties of the bio composites.

Other than that, related to the finding, the alkaline treated coconut husk fibre has affected the density of the bio composite. The implementation of NaOH treatment in removing the less dense elements from the coconut husk fibre such as hemicellulose and lignin that are one of the factors that contributed to the high-density percentage for the alkaline treated sample. This phenomenon also been mentioned in a study by Ouarhim et al. (2019). Following that, an investigation by Pokhriyal et al. (2023) stated that during the alkaline treatment, the molecules that attached to the fibre surface also filled in the groove. Besides, the element of cellulose has high molecular weight whereas it increased the bio composite's density value.

Table 4 below shows the density analysis of variance result for both treated and untreated sample. Additionally, the result of ANOVA shows the P-value for both of untreated and treated sample were lower than 0.05. This indicates that there are significant variations in the mean of density between the various ratios of the bio composite.

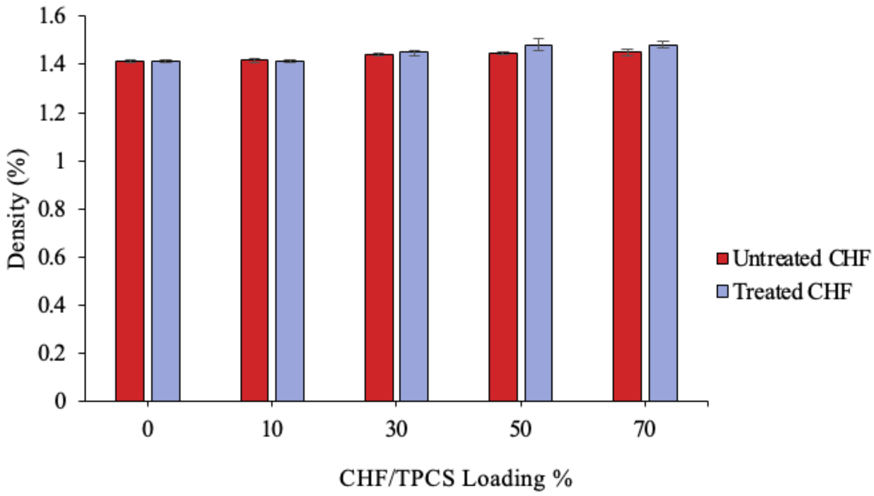

FIGURE 14. The density of the untreated and alkaline treated samples.

TABLE 4. The density analysis of variance.

Type of sample	df	P-value Density
Untreated sample	4	1.0375×10^{-3}
Treated sample	4	1.66216×10^{-4}

X-RAY DIFFRACTION (XRD) ANALYSIS

Table 5 below displayed a table of crystallinity index percentage for pure coconut husk fibre, NaOH treated coconut husk fibre and various samples. This data indicated the samples' structure and crystallinity improvement responding to the alkaline modification.

First and foremost, for the composition between the coconut husk fibre content and the thermoplastic cornstarch, this outcome that found that the rises number of fibre loading in the samples has increase the value of crystallinity index. This finding is related to the statement that has been mentioned in a study that has been conducted by Zubairi et al. (2023) that stated the number of fibre loading created the nucleation effect which also contributed to the increment and the enhancement of the crystallinity in the bio composite.

Other than that, the table display the details of the crystallinity index of pure coconut husk fibre is 39.93% whereas the alkaline treated coconut husk fibre crystallinity index is 83.46%. The crystallinity index of alkaline treated coconut husk fibre is higher than the pure coconut husk fibre. In addition, the value of crystallinity index between the samples is significantly different. The pure thermoplastic shows the lowest crystallinity index with the number of 22.03% while the highest one is at 30% of alkaline treated composite with the value of 95.89%.

This investigation also found that the alkaline treated composite for all samples have greater number in crystallinity index compared to untreated sample. However, the crystallinity index at 70% for both untreated and alkaline treated samples is slightly different which is 2.41% while between 30% of both untreated and alkaline treated samples has wide differences which is 54.8%. This proved that the alkaline treated coconut husk fibre has enhanced the structural and properties of the bio composite.

Overall, the alkaline treated coconut husk fibre has enhanced the crystallinity in the bio composite. This finding of the improved crystallinity may result a good mechanical properties and thermal properties. In addition, it is also has been proved by the tensile testing whereas the alkaline treated sample has improved the tensile properties. Other than that, this alkaline treated coconut husk fibre also attributed to the durability of the bio composite. This statement also been mentioned in an investigation that has been conducted by Kotzur et al. (2022) whereas it said the improvement of the crystallinity indicate that the polymer becomes more rigid which the improved polymer has high melting point, high thermal resistance and this characteristic has displayed that the polymer has a good interfacial strength between matric and fibre and have an excellent toughness.

TABLE 5. XRD data is showing the treated sample has high crystallinity index over untreated sample.

Sample	CI%
Untreated CHF	39.93
Treated CHF	83.46
0% TPCS/CHF	22.03
10% TPCS/CHF	27.51
10% TPCS/TCHF	90.92
30% TPCS/CHF	41.09
30% TPCS/TCHF	95.89
50% TPCS/CHF	75.04
50% TPCS/TCHF	89
70% TPCS/CHF	82.74
70% TPCS/TCHF	85.15

THERMOGRAVIMETRIC ANALYSIS (TGA)

Figure 15 below displays thermogravimetric curve of untreated bio composite and alkaline treated bio composite using TGA analysis.

From the thermogravimetric curve above, the results of all samples unveiled various curves that can be an indication of thermal properties of the bio composites. First and foremost, the curve of the testing indicates that the mass loss of the bio composites is due to the increment of the temperature. At the ranging temperature between 25°C to 120°C, this is the first phase of the degradation reaction happened to the samples. In this phase, pure thermoplastic cornstarch showing the major drop of mass compared to other samples while the untreated samples of 70% fibre content reacted at the slowest rate. This first phase is the phase where the water and any other moistures in the samples escaped and evaporated as mentioned by Sivakumar et al. (2022).

Furthermore, the second phase of degradation occurred between 130°C to 340°C. This phase is the highest percentage of mass loss to happen and become the phase where the fastest degradation rate occurred. In this phase, some of the natural elements namely cellulose, hemicellulose and lignin exposed to the thermal degradation. This can be proven with the statement in a research paper written by Hafila et al. (2021).

Other than that, the maximum temperature of the bio composites decomposition happened at 340°C whereas it can be attributed to the degradation of thermoplastic cornstarch as quoted by Diah et al. (2021). Finally, the final phase happened at the temperature of 360°C until 600°C. In this phase, the mass of the bio composite slowly reduced which indicated the ash formation starting to take place and the next increment of the temperature will continue for further decomposition. At this point, the TGA curves unveiled that the lower residual mass happened to pure thermoplastic cornstarch compared to the thermoplastic cornstarch that reinforced with coconut husk fibre. Besides, the alkaline treated shows the better in thermal resistance by observing at the residual mass in contrast with the untreated sample. From this analysis, the data of the thermal properties has been collected.

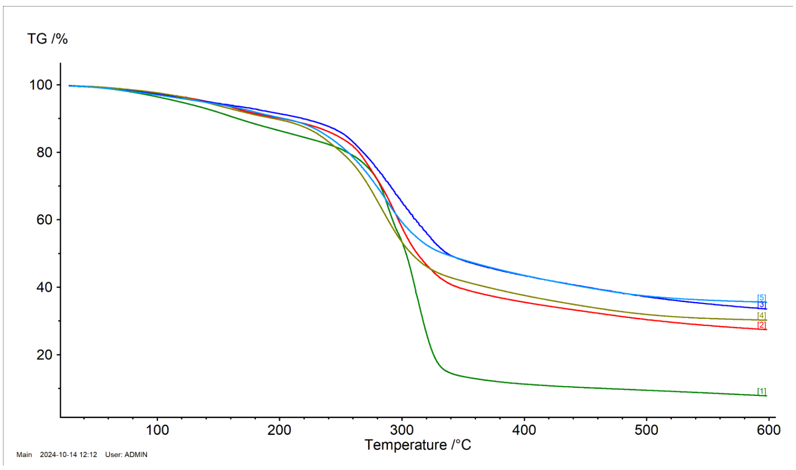

FIGURE 15. The thermogravimetric curve of the untreated and alkaline treated samples.

Table 6 below contains important finding that become several significant factors for analysing thermal degradation rate, thermal resistance and thermal stability of the untreated samples and alkaline treated sample.

The table 6 below portrayed significant data that indicate the thermal properties of both untreated and alkaline treated samples. First and foremost, the control samples of pure thermoplastic cornstarch show a higher mass changes by 92% from the initial weight. The mass changes percentage constantly dropped responding to the different types of samples. In details, untreated sample at 50% and alkaline treated sample at 50% unveil the mass changes values of 72.31% and 69.63% respectively.

Following that, at 70% fibre content for both untreated and alkaline treated samples show the mass changes percentage of 66.63% and 64.15% respectively. Besides, the table also displayed the percentage of the residual mass which incline as the fibre content increase, and it also responded to the variable of alkaline treatment whereas the alkaline treated samples have higher residual mass percentages compared to the untreated samples for both 50% and 70% coconut hush fibre loading.

According to the table, the onset temperature also differs between the samples, the highest onset temperature is at 70% fibre content of alkaline treated samples with the temperature of 253.2 °C. In contrast, the lower onset temperature is 123.6 °C that has been obtained from pure thermoplastic cornstarch.

The thermal characteristics of various ratios of coconut husk fibre in bio composite has been analysed with the result obtained. The outcomes display that the greater coconut husk fibre loading can resist high temperature. It can be proved that with the addition of coconut husk fibre increases has firmly affect the thermal properties whereas the higher fibre loading indicates a better residual mass as well as thermal resistance of the bio composite. This is related to a study by Shah et al. (2021) where it quotes the improvement of thermal degradation might be attributed by the natural elements' thermal crosslinking that naturally presence in coconut husk fibre.

Furthermore, the obvious differences of TGA analysis between the untreated samples and the alkaline treated samples is related to the modification of the fibre. In details, the alkaline treatment by using NaOH has eliminated the amorphous element in the coconut husk fibre. Therefore, the thermal stability of the alkaline treated sample is better than the untreated samples. This phenomenon indicated that the alkaline treated samples have high thermal deprivation resistance after the impurities and unwanted elements have been discarded from the coconut husk fibre, this finding is also stated by Bharath et al. (2022).

Other than that, a study that has been conducted by Kamaruddin et al. (2022) mentioned that the coconut husk fibre undergoes alkaline treatment has positively affect the behaviour of the thermal degradation on the bio composite whereas it has proven by the percentage of the mass residual for the alkaline treated samples were higher than the untreated samples. This could lead to an excellent and stable thermal of the bio composite.

Besides, the alkaline treated sample does not show great mass loss in the range of temperature which proved the enhancement of the thermal degradation stability. Related to this, one of the main factors is the minute number of lignin and hemicellulose after the alkaline treatment that directly remove the non-cellulose element. This finding was correlated with a study that has been made by Abdullah et al. (2024). This outcome exposes that the alkaline modification on the coconut husk fibre has also modified the surface and the thermal properties of the fibre which led to high resistance to thermal and have better thermal stability compared to the pure coconut husk fibre, other than that, the ratios of the fibre also play an important role for the thermal properties' enhancement for the bio composite.

TABLE 6. TGA data showing the treated sample has high thermal stability than untreated sample.

Sample	Onset temperature (°C)	Residual mass (%)	Mass changes (%)
0%	123.6	7.80	92
UT50%	196	27.44	72.31
T50%	229	30.23	69.63
UT70%	247.8	33.55	66.63
T70%	253.2	35.55	64.15

CONCLUSION

In summary, this investigation was utilizing alkaline treated coconut husk fibre by using NaOH as the reinforcement in thermoplastic cornstarch to produce bio composite. This investigation undergoes several analyses for the evaluation of mechanical, physical and chemical properties between the alkaline treated samples and untreated samples whereas the analyses highlight that the alkaline treatment does enhance the properties of the bio composite. In term of tensile properties, the alkaline treated sample illustrated a better result in tensile strength than the untreated sample by the differences of 1.34 MPa, 0.58 MPa, 2.54 MPa, and 2.85 MPa of each ratio. Other than that, for the hardness characteristics of the bio composite in this research also highlight the alkaline treated samples have improved hardness resistance than the untreated sample. In SEM, the image of the result showed the binding between fibre and thermoplastic cornstarch of the alkaline treated samples is strong and completed compared to the untreated samples.

In addition, for density properties, the sample also show the same trends whereas the alkaline treated sample increased the density number compared to untreated samples by the percentage differences of 0.005%, 0.008%, 0.035% and 0.031% for each ratio. This is due to the elimination of low-density elements such as lignin and wax of the coconut husk fibre. As for moisture content testing, alkaline treated sample contained low moisture than the untreated samples, this is due to high hydrophilicity of the natural coconut husk fibre which it contains hydroxyl group elements while the treated coconut husk fibre improved the binding avoiding the moisture to enter the interface of the bio composite. In XRD, the crystallinity index for alkaline treated samples is higher compared to untreated samples. This can be a factor for the enhancement of the mechanical properties of the bio composite which it also shows alkaline treated coconut husk fibre has narrow intensity indicating the sample has high crystallinity and has low number of amorphous elements such as lignin and hemicellulose. In TGA, the fibre content played a role which affected the ability to resist thermal exposure. This is because the high percentages fibre content samples show the higher number in the residual mass compared to the lower fibre content samples. In instance, for the sample of 50% of fibre shows 27.44% and 30.23% for both untreated and treated samples, compared to the higher fibre content of 70% for both untreated and treated sample, the residual mass is 33.55% and 35.55%. The TGA analysis indicated that the alkaline treatment has eliminated amorphous element in the coconut husk fibre and has improved the thermal stability of the bio composites. These findings may contribute to the innovation of food and beverages industry whereas the durability in mechanical, physical and thermal of the sample is giving a promising potential for the fabrication of food and beverage container such as tray, cup, straw or plate. In future, the study can be continued for its fabrication ability and its machinability.

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DECLARATION OF COMPETING INTEREST

None.

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