Journal of Engineering and Technology

ISSN 2180-3811 eISSN 2289-814X https://journal.utem.edu.my/index.php/jet/index

CHARACTERIZATION OF COTTON FIBER FILLED POLYVINYL ALCOHOL FILMS

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Article history:
Received Date: 2021-09-24
Accepted Date: 2021-12-21

Keywords:
Biodegradable, Cotton fiber, Polymer films, Polyvinyl alcohol (PVA), Tensile modulus

Abstract— Petroleum-based polymer films have high resistance toward degradation and bring about countless environmental problems due to inadequacy of recycling and low degradability. To counter these problems, it can be conquered with the development of biopolymer by adding organic fibers, which makes the films to be more biodegradable. In this study, cotton fiber-filled polyvinyl alcohol (PVA) films were prepared with various cotton fibers loading, which is 0, 5, 10, 15, and 20 wt% in 20g, 25g, and 30g of PVA. It is aimed to test the tensile properties, hardness, and thermal stability of the PVA/cotton fiber blended films. The surface morphology and surface fracture of the blended films were conducted by using

scanning electron microscopy. Based on the result obtained, PVA/cotton blended films with higher cotton fiber loading demonstrated lower tensile strength and elongation at the break due to the weak intermolecular bonding between the cotton fiber and the PVA matrix. However, the tensile modulus was improved gradually as the increment of cotton fiber loading resulted in stiffer blended films as compared to pure PVA film. The hardness value of the PVA/cotton fiber shows a significant boost when the cotton fiber is added as filler into the PVA matrix as well. In conclusion, the study shows that PVA/cotton fiber blended film has stiffness that surpasses the hardness values of brass, soft steel, and aluminum, in which it has a higher potential to replace metals as in home construction in the future.

I. Introduction

In the first half of the 20th century, PVA has been applied in the areas such as industrial, medical, food, and commercial sectors, which are being used to generate several end products, which include resins, lacquers, surgical threads, and food packaging supplies [1]. PVA as if a promising source for food packaging due to its characteristics, which have decent flexibility, durability, transparency, biocompatibility, toxic-free, barrier properties, and biodegradability [2]. Polymer composite has worthy potential to be utilized in the construction sector. Many constructors equip fiber-reinforced polymer (FRP) in construction, which include bridges, masonry walls, tanks,

foundations, and buildings. FRP was also applied in the refurbishing and rehabilitation of conventional building materials that consist of concrete, steel, masonry, and wood [3]. In this study, cotton fiber is to be incorporated into the production of PVA/cotton fiber composites of different wt% and PVA by weight. The samples are subjected to Field Emission Scanning Electron Microscopy, Thermogravimetric analysis, Rockwell Hardness, and Mechanical properties tensile strength tests. Cotton fiber acted as the reinforcement in the PVA matrix can offer remarkable mechanical properties as in tensile modulus and the flexural modulus. The method fortifies the stiffness and hardness of PVA where it can be an alternative for home construction materials in the future [4].

II. Methodology

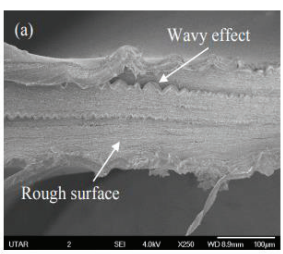
Three sets of PVA solutions were prepared by dissolving 20, 25, and 30g of PVA powder respectively in 200ml of distilled water in the 100°C water bath with a magnetic bar at a constant stirring rate (2 RPM) for 4 hours until and a clear colorless solution was obtained. The prepared PVA solution was then poured into a 7cm x 7cm cotton fabric with different cotton fiber loading, which is 0, 5, 10, 15, 20 wt% for coating processes. The cotton was padded in the PVA solution. The finishing film was dried overnight at 70°C. The morphology of the films was analyzed using a field emission scanning electron microscope (FESEM), model JOEL JSM 6710F. The films were coated with platinum to enhance electron charging during imaging processing. The hardness of the films was tested using a model CV-600MBD Rockwell Hardness Tester, which compiled to ASTM E-18 standard. Tensile testing on films was carried out using a model H10KS-0748 light-weight tensile tester, from Tinius Olsen. The films were cut into dumbbell shapes with a gauge length of 26mm and the next width of 3mm for testing.

III. Results and Discussion

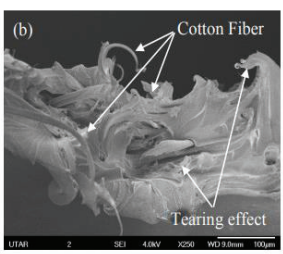
A. Field Emission Scanning Electron Microscopy (FESEM) Analysis

Figure 1 shows the 250X low magnification power images of the 20g unfilled PVA and various compositions of PVA/cotton fiber, which are 20g/10 wt%, 20g/20 wt%, 25g/10 wt%, 25g/20 wt%, and 30g/20 wt%. Figure 1(a) shows that the unfilled PVA has the roughest surface as compared to various blended films, which are shown in Figure 1(b) to Figure 1(*f*). The rough surface is being more ductile due to the contribution of the good matrix tearing caused by the highest mobility of the PVA chains. As a result, the high deformability of the unfilled PVA allows more elongation when stress is applied. This is compatible when the data obtained where the elongation at break for the unfilled PVA of 20g is being the highest at 293.8% before breakage. Meanwhile, there is a wavy effect can be observed on the pure PVA in which this wave-like structure is formed due to the rebounding effect of the elongated PVA matrix as it breaks during the stretching process. In short, the presence of wave-like structure in the unfilled PVA film shows that it has greater elongation under plastic deformation as it is subjected to strain where this is compatible with the data obtained in the tensile strength. Moreover, the adhesion of cotton fiber as filler to the matrix of PVA is observed in Figure 1(b) where 10% of cotton fiber loading is implied in 20g PVA. Based on the figure, there is a lower surface area available for the adhesion to occur as compared to the unfilled PVA where this may be leading to the poor adhesion of PVA and cotton fiber. Henceforth, to improve the interactions between filler and matrix, smaller particles size of the fillers is always preferred [5]. Meanwhile, the tearing effect can be noticed where this may be caused by the poor dispersion of cotton fiber that then weakens the effect of interfacial adhesion as well as the interaction with the polymer matrix. Therefore, it can be said that the poor

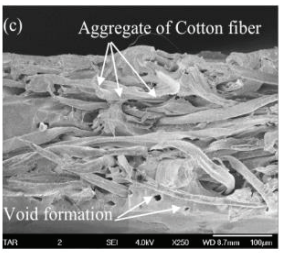
dispersion of cotton fiber increases the weak point or stress concentration point in which causes the PVA composites to break at the stress concentration point and hence, lowers the tensile strength. Filler aggregation can be observed in Figures 1(c), (d), (e), and (*f*) especially when the cotton fiber loading increases to 20%. The aggregation of cotton fiber, which acts as the filler, will bring about poor dispersion and non-uniform distribution of cotton fiber in the matrix of PVA [6]. The cotton fiber fillers that were being coarsely dispersed may be the main reason for the poor filler-matrix interaction poor filler-matrix interaction whereas this will then diminish the performance of the PVA/cotton fiber blended film on parameters that include both the tensile strength and break elongation. Furthermore, the aggregation of the cotton fiber had transformed the blended films to become more brittle while the tensile modulus for the increasing loading of cotton fiber blended films becomes higher. Meanwhile, the crazing effect may also be incurred by the aggregation of cotton fiber where the adhesion of the cotton fiber fillers with the PVA matrix phase is ruined [7].



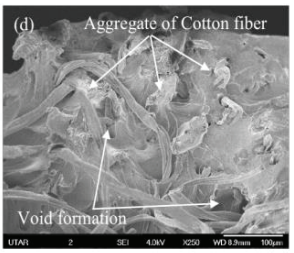
(a) PVA/cotton fiber 20g / 0 wt%



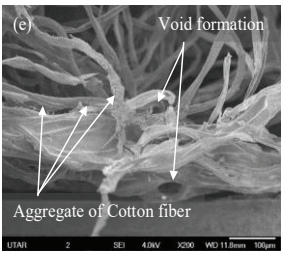
(b) PVA/cotton fiber 20g / 10 wt%



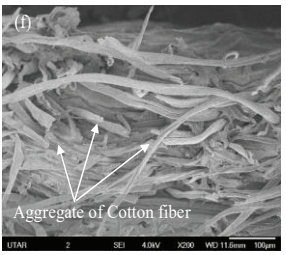
(c) PVA/cotton fiber 20g / 0 wt%



(d) PVA/cotton fiber 25g / 10 wt%



(e) PVA/cotton fiber 25g / 20 wt%

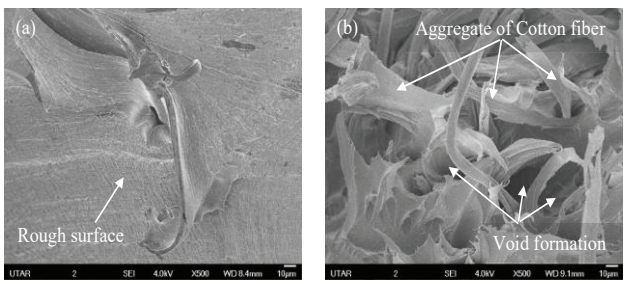


(f) PVA/cotton fiber 30g / 12 wt%

Figure 1: Comparison of surface fracture of PVA/cotton fiber at 250X low magnification power

By observing results shown in Figure 1(c), (d), and (e), there are void or holes which are also known as cavities are noticeable especially in PVA/cotton fiber composite with 20g/20 wt%, 25g/10 wt%, and 25g/20 wt%. The formation of voids could transform into weak points of the PVA composites where the cavities will become the stress concentration point which causes it to be torn apart when it is subjected to strain. Therefore, the formations of cavities in the PVA/cotton fiber composites as shown in the figures have high tendencies in leading to films with low tensile strength and elongation at break. The clearer SEM images analyzed by using the higher magnification of 500 are shown in Figure 2. Based on the figures, the aggregations of cotton fiber will form the flaws in the film [8]. Meanwhile, the flaws will have a high tendency in propagating into voids when

there is increasing in size, contributing to the poor interfacial interaction. Furthermore, the flake-like structure can be observed in Figure 2(c). The existence of the flake-like structure is attributable to the non-homogeneous dispersion of cotton fiber in the matrix of PVA in which this is then weakening the interfacial adhesion effect and the interaction with the PVA matrix. Therefore, the flake-like structure presented on the cotton fiber may act as a lubricant as in the PVA matrix especially when the moment PVA/cotton fiber composite is experiencing elastic deformation which is subjected to straining. Consequently, the flake-like structure in PVA/cotton fiber blended film will return to lower tensile strength.



(a) PVA/cotton fiber 20g / 0 wt%

(b) PVA/cotton fiber 20g / 10 wt%

(c) PVA/cotton fiber 20g / 20 wt%

(d) PVA/cotton fiber 20g / 30 wt%

Figure 2: Comparison of surface fracture of PVA/cotton fiber at 500X high power magnification

B. Thermogravimetric Analysis (TGA)

Figures 3, 4, and 5 show the TGA curves of various cotton fiber loadings in 20g, 25g, and 30g of PVA. Based on the figures, there shows a similar trend where all the samples experienced a weight loss of around 10% within the temperature of 30°C to 300°C which is due to the evaporation of moisture contents.

The region between temperature 300 and 420°C is associated with the decomposition and carbonization of the PVA where the polymer started to undergo thermal degradation due to the deterioration of PVA polymeric molecules and the components of the long-chain backbone of the PVA experiences chain scission and may react with one another to alter the properties of the polymer. Figures 6, 7, 8 9, and 10 show the comparison of TGA plots of different cotton fiber loading in various PVA compositions. Based on the results, the region between temperature 30 and 250°C is due to the loss of water. From 250 to 300°C, the bonds in cotton fiber break while the main PVA chains undergo cleavage between 300 and 450°C. It is observable that the temperatures of the weight loss regions demonstrated a small increment with the cotton fiber content, indicating that cotton fiber increases the thermal stability of the PVA/cotton fiber membranes at high temperatures.

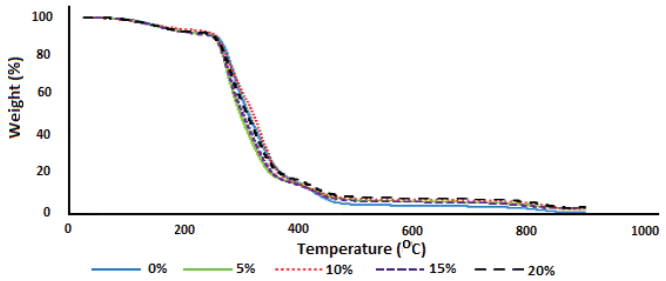


Figure 3: TGA plot of different cotton fiber loading in 20g of PVA

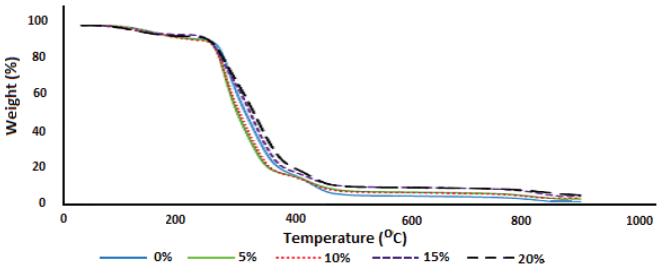


Figure 4: TGA plot of different fiber loading in 25g of PVA

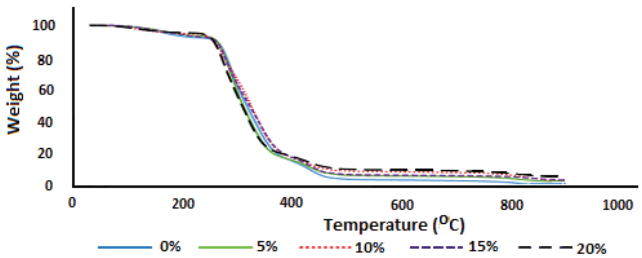


Figure 5: TGA plot of different cotton fiber loading in 30g of PVA

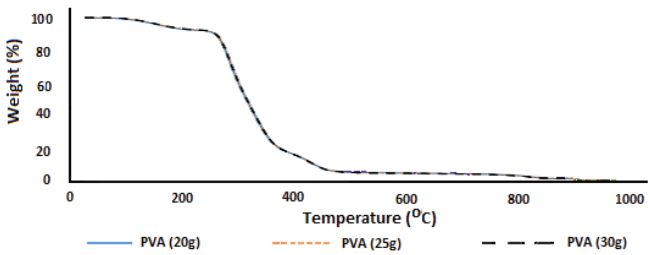


Figure 6: TGA plot of 0 wt% cotton fiber loading in various PVA composition

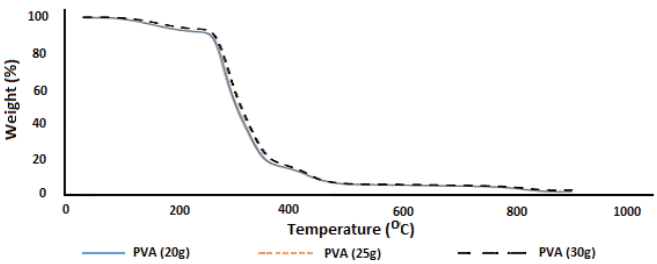


Figure 7: TGA plot of 5 wt% cotton fiber loading in various PVA Composition

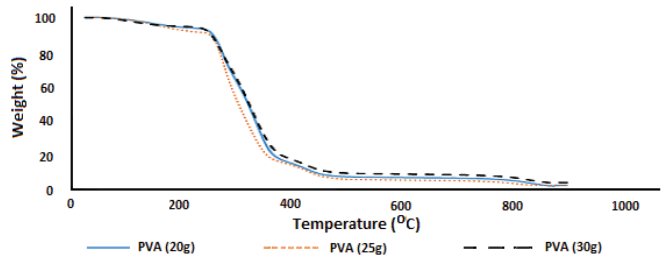


Figure 8: TGA plot of 10 wt% cotton fiber loading in various PVA composition

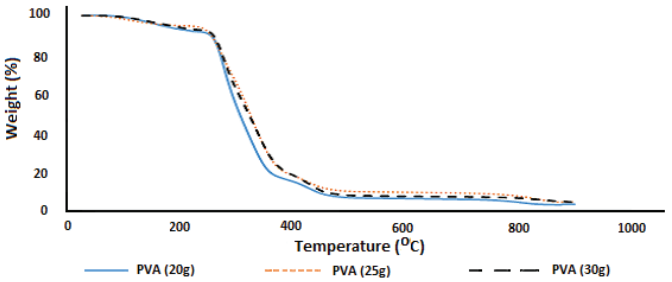


Figure 9: TGA plot of 15 wt% cotton fiber loading in various PVA composition

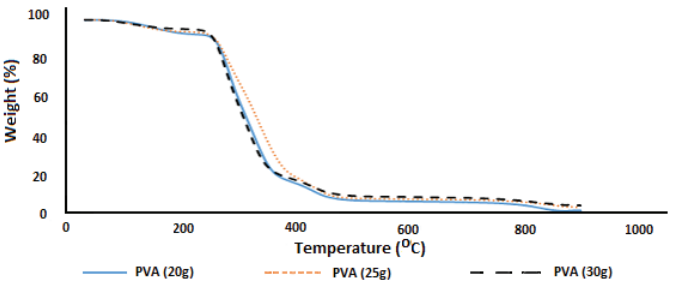


Figure 10: TGA plot of 20 wt% cotton fiber loading in various PVA composition

C. Rockwell Hardness Test

The Rockwell Hardness values (HRB) of the blended films with various compositions of PVA/cotton fiber are shown in Figure 11 to Figure 17. They show a growing trend as of the hardness values of the PVA/cotton fiber composites as the loading of cotton fiber increases. The hardness of PVA can be explained as the property of the PVA matrix, which enables it to resist the plastic deformation, by penetration or by indentation where this may be referred to the stiffness or temper, or resistance to bending,

scratching abrasion, or cutting of the produced PVA films. As the loading of cotton fiber increases, it improves the ability of the composite films to resist being permanent, deformed when there is a heavier load is applied. This is due to the higher cotton fiber loading inducing the greater formation of hydrogen bonding and interfacial adhesion between the PVA matrix and cotton fiber. This may then play a vital role in hindering the mobility of the PVA chains when it is subjected to stress and strain. This grants the blended films greater hardness values as there will be more force or stress required to cause the PVA/cotton fibers composite films to undergo deformation. The greater the hardness of the material, the better the resistance that may tackle deformation. Meanwhile, the addition of cotton fiber, which may act as a reinforcing agent within the PVA matrix, increases the rigidity of the PVA composites. Therefore, the hardness of the film increases as the rigidity of PVA/cotton fiber composites increases.

Based on Table 1, the highest Rockwell Hardness value obtained is 98.4 HRB when there is 20 wt% cotton fiber loading being incurred in 25g of PVA where this formulation of PVA/cotton fiber blended film may exhibit the best performance as in hindering the flow of PVA chain, which makes it to gain the highest stiffness among all. It is comparable with HRB value shown in Table 2, for a few types of metals which is commonly used in home construction such as the metal frame for windows and fences.

It is found that the values of a few sets of PVA/cotton fiber composition which include 25g/20 wt%, 30g/10 wt%, and 30g/15 wt% are way higher than the brass, soft steel, and aluminum which gives the composites a higher potential to replace metals as in home construction in the future.

Table 1: Rockwell hardness value (HRB) of different Composition of PVA/cotton fiber

Cotton Fiber Loading (wt%)	PVA 20g	PVA 25g	PVA 30g
0.0	20.9	66.0	78.6
5.0	29.1	69.8	90.6
10.0	30.1	72.8	98.4
15.0	38.9	76.5	86.4
20.0	87.8	90.9	79.5

Table 2: Rockwell Hardness values (HRB) of metals commonly use in home construction
Source: MatWeb Material, Property Database (2020)

Type of Metal	Rockwell Hardness Value (HRB)
Brass	67.4
Soft Steel	88.5
Aluminium	75.0

D. Mechanical Properties

Tensile Strength Test

Figures 12, 13, and 14 demonstrate the analysis of the ultimate tensile strength and elongation at break for different loading of cotton fiber which ranges from 0% to 20% incorporated in different PVA content which ranges from 20g to 30g whereas Figures 15 and 16 compare the ultimate tensile strength and elongation at break of films with different composition of PVA/cotton fiber respectively. Based on the results, both parameters exhibit similar trends where the tensile strength and the elongation at break are declining as the loading of cotton fiber increases.

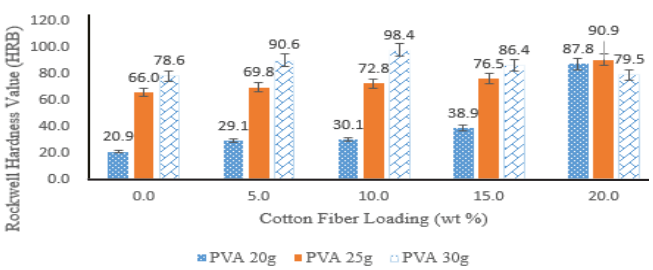


Figure 11: Rockwell Hardness values (HRB) of films with different compositions of PVA/cotton fiber

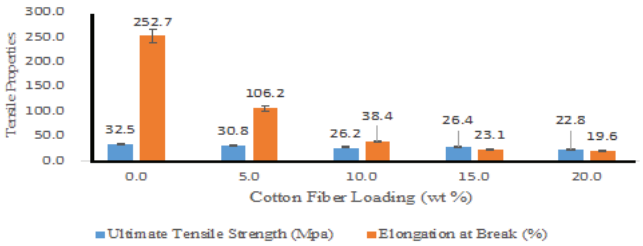


Figure 12: Tensile properties of films with different cotton fiber loading in 25g of PVA

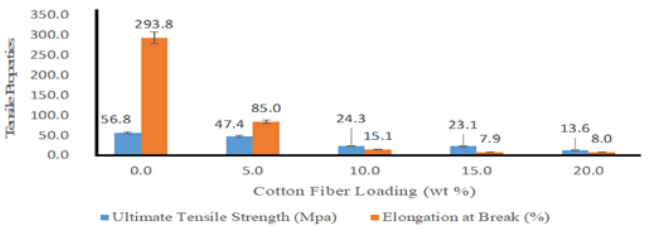


Figure 13: Tensile properties of films with different cotton fiber loading in 20g of PVA

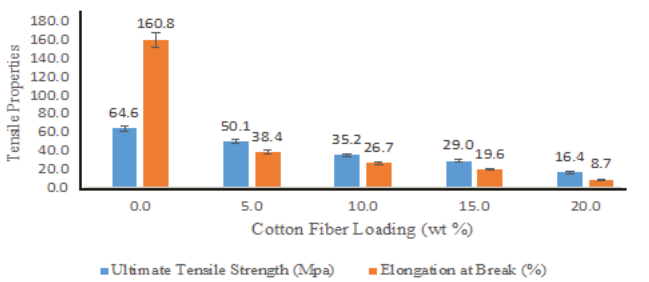


Figure 14: Tensile properties of films with different cotton fiber loading in 30g of PVA

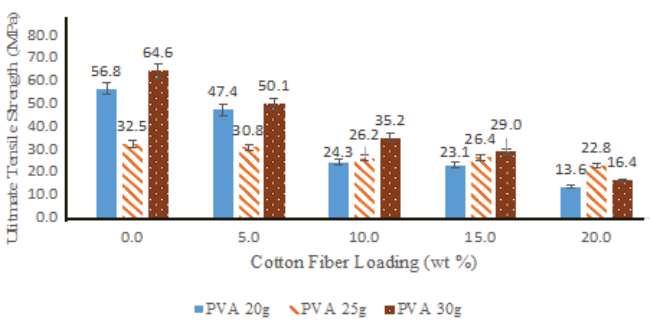


Figure 15: Comparison of ultimate tensile strength of films with different compositions of PVA/cotton fiber

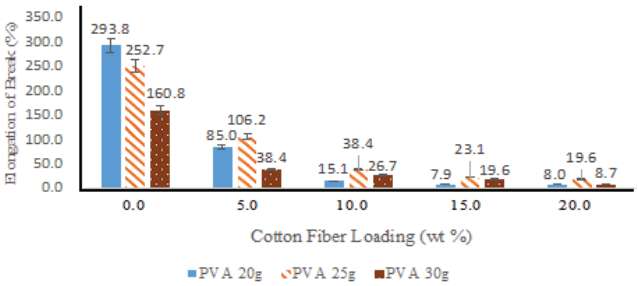


Figure 16: Comparison of elongation at break of films with different compositions of PVA/cotton fiber

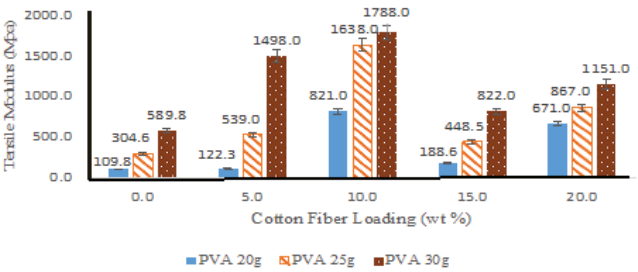


Figure 17: Comparison of tensile modulus with different compositions of PVA/cotton fiber

Noticeably from Table 3 and Table 4, the tensile strength and elongation at break of the unfilled PVA are the highest in all cases followed by the increment of the cotton fiber loading from 5 to 20 wt %. This may be due to the reduction of the PVA matrix content in place of the PVA exhibiting the ductility properties [8]. This may also be due to the improper dispersion of cotton fiber in PVA, which brings about the agglomeration of the cotton fiber and weakens the tensile strength of the composites by decreasing the bonding interaction. The agglomerated fibers may act as the stress concentration point during straining where the stress is unable to be transferred to the PVA matrix effectively as well.

Meanwhile, the low tensile strength and the elongation at the break of the composites might be attributable to the weak intermolecular hydrogen bonding between the cotton fiber filler and PVA matrix. The

bonding between the PVA and cotton fiber tends to become weaker for the higher loading of cotton fiber. This is compatible with the research conducted by [9], where PVA/banana front flour blended films presented a much lower tensile strength as the use of higher loading of banana front flour. Based on Figure 11 and Table 3, the PVA/cotton fiber blended films demonstrate higher tensile modulus with the increase of cotton fiber loading as compared to the unfilled PVA in all cases. The incorporation of cotton fiber which acts as fillers in the films will restrict the mobility of the PVA chains when it is subjected to force which is then increasing the brittleness of the blended films.

Table 3: Ultimate tensile strength (Mpa) of films with different compositions of PVA/cotton fiber

		Cotton Fiber Loading (Wt %)				
		0.0	5.0	10.0	15.0	20.0
PVA	20g	56.8	47.4	24.3	23.1	13.6
	25g	32.5	30.8	26.2	26.4	22.8
	30g	64.4	50.1	35.2	29.0	16.4

Table 4: Elongation at break (%) of films with different compositions of PVA/cotton fiber

		Cotton Fiber Loading (Wt%)				
		0.0	5.0	10.0	15.0	20.0
PVA	20g	293.8	85.0	15.1	7.9	8.0
	25g	252.7	106.2	38.4	23.1	19.6
	30g	160.8	38.4	26.7	19.6	8.7

Based on the data obtained, the tensile modulus of the PVA composites exhibited a significant increment given that when the cotton fiber loading is increased to 10%, which can be described as the optimal among all loadings of cotton fiber. This may be due to the addition of 10% cotton fiber as filler which can induce the maximum formation of hydrogen bonding

and interfacial adhesion between the PVA matrix and cotton fibers where this may be the best performance in hindering the mobility of the PVA chains when it is subjected to force. Moreover, the addition of cotton fiber, which may act as a reinforcing agent within the PVA matrix, is increasing the rigidity of the PVA composites. Therefore, the tensile modulus increases as the rigidity of PVA/Cotton fiber composites increases. However, the tensile modulus of the PVA composites in 15% and 20% of cotton fiber loadings showed a drop as compared to the 10% cotton fiber loading. This may be owing to the higher amount of cotton fiber used which is then causing the poor dispersion of cotton fiber, which may then form an agglomerate. The agglomeration of cotton fibers is said to weaken the interfacial adhesion between the filler and PVA matrix which in turn lowers the rigidity of the PVA/cotton fiber composites. Consequently, the tensile modulus shows decrement when 15% and 20% of cotton fiber loadings are used.

IV. Conclusion

Cotton fiber-filled PVA films were successfully produced at 0, 5, 10, 15, and 20 wt% of cotton fiber loading with 20g, 25g, and 30g of PVA. The surface morphology of the PVA was found to be smooth and fine where it contributes to the transparency properties. Based on the data obtained, the incorporation of the cotton fiber in the PVA demonstrated a negative effect on the tensile strength and elongation at break. While the tensile modulus shows a growing trend with the increasing of the cotton fiber loading from 5-20 wt% as compared to pure PVA in all contents. In the surface fracture analysis, aggregation of cotton fiber can be observed especially at the higher loadings of cotton fibers from 5 to 20 wt% which causes the formation of voids where the poor filler/matrix interaction would be the consequence in contributing to the lowering of ultimate tensile strength of the blended films as the cotton fiber loadings increases. Meanwhile, it is noticeable that the addition of cotton fiber as filler in the PVA

matrix can slightly increase thermal stability. Moreover, the addition of cotton fiber is shown to be able in fortifying the PVA matrix, causing the films to be harder and stiffer as the cotton fiber loading is going up to 20 wt%. The highest Rockwell hardness value obtained is 98.4 HRB for 20 wt% cotton fiber loading 25g of PVA. This formulation of PVA/cotton fiber blended film where such stiffness surpasses the hardness values of brass, soft steel, and aluminum. It gives the composites a higher potential to replace metals as in home construction in the future.

V. References

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