

An Initial Study on the Effects of Dolomite on the Tensile Properties of Agar/Kenaf Biocomposite Films

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Abstract: In this initial study, agar/kenaf core fiber (KCF) biocomposite films, with and without dolomite (Dol), were prepared by solution casting. The prepared biocomposite films were cut into a rectangular shape for tensile testing. The tensile test of the biocomposite films was conducted using a universal testing machine. The tensile test results indicated that the incorporation of KCF increased tensile strength at 4 wt.% KCF, while Young's modulus increased at 12 wt.% KCF. However, elongation at break decreased progressively as KCF content increased. Based on the tensile strength, 4 wt.% KCF was selected for Dol incorporation. Incorporating Dol resulted in the highest tensile strength and Young's modulus at 4 wt.% Dol, accompanied by decreased elongation at break. Statistical analysis revealed that KCF content significantly affected all tensile properties, whereas Dol content significantly affected all tensile properties except tensile strength. In summary, KCF and Dol incorporation increased the stiffness and tensile performance of agar-based biocomposite films while reducing their extensibility.

Keywords: Kenaf core fiber, Solution casting, Tensile strength, Elongation at break, Young's modulus.

1. INTRODUCTION

Biocomposite films have attracted attention as sustainable materials because they combine renewable biopolymers with natural reinforcing substances to fabricate environmentally friendly alternatives to traditional petroleum-based films [1]. These materials are developed from polysaccharides, proteins, or other biodegradable polymers, which are incorporated with plant-derived fibers, particles, or bio-based reinforcements [2, 3]. The combination of these materials can improve the structural and functional properties of the resulting films while maintaining their biodegradability [4]. Biocomposite films are particularly promising for applications in packaging, coatings, agriculture, and other disposable products where continued reduction of plastic waste is increasingly important. Their properties are strongly influenced by the type and loading of the matrix and reinforcing agent, as well as their dispersion and interfacial interactions [5]. Consequently, careful preparation and processing are essential for producing homogeneous films with desirable performance. Continued research on biocomposite films therefore supports the development of sustainable materials from renewable resources for practical applications.

Agar is a natural polysaccharide derived from marine algae and is regarded as a promising matrix for biocomposite films due to its film-forming ability, renewability, biodegradability, and availability [6]. Figure 1 shows the chemical structure of agar. Nevertheless, agar-based films may require reinforcement to improve their performance and broaden their applications. Kenaf core fiber is a renewable lignocellulosic material containing cellulose, hemicellulose, and lignin [7, 8]. Incorporating it into an agar matrix may reinforce the film while promoting the utilization of plant-based resources. The tensile properties of biocomposite films are important indicators of their ability to withstand applied forces during use and handling. These properties are assessed through tensile strength, elongation at break, and Young's modulus, which represent resistance to failure, flexibility, and stiffness, respectively [9]. The resulting tensile behavior depends on fiber content, fiber dispersion, and matrix-fiber interfacial adhesion, which determine the effective stress transfer between the agar matrix and the kenaf core fiber.

Dolomite is a naturally occurring mineral primarily composed of calcium magnesium carbonate and can serve as a secondary filler in biocomposites without surface treatments of the natural fibers [10]. Its incorporation is also in high demand as a co-filler in hybrid polymer composite systems. Dolomite particles can enhance the physical and mechanical properties of polymer composites in ways not achievable with a single filler alone [11]. The effectiveness of dolomite

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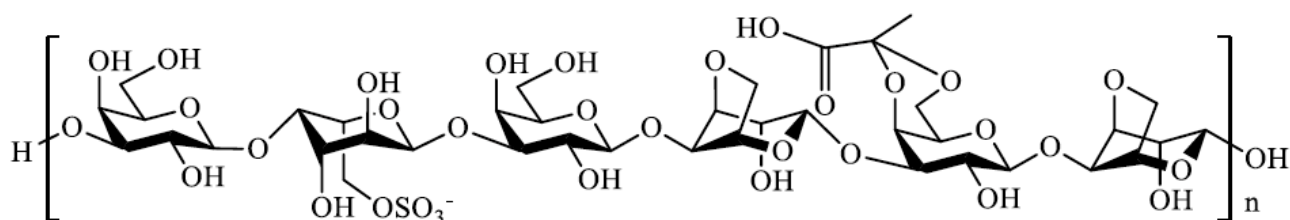


Figure 1: Chemical structure of agar.

depends on factors such as particle size, loading, dispersion, and interfacial adhesion with the surrounding matrix. Therefore, incorporating dolomite into biocomposite films provides a means to tailor material properties while utilizing an abundant mineral resource. In this initial study, dolomite was incorporated into agar/kenaf biocomposite films. Unlike previous studies focusing primarily on polymer/kenaf biocomposites, this study introduces dolomite as an additional mineral filler and evaluates its contribution to the tensile performance of the biocomposite films. This study therefore provides initial information on the combined use of kenaf core fiber and dolomite as reinforcements in an agar-based matrix. The objective of this study is to investigate the effects of kenaf core fiber and dolomite on the tensile properties of agar-based biocomposite films. The biocomposite films with improved tensile properties could potentially be used to produce various products.

2. MATERIALS AND METHODS

2.1. Materials

Agar and glycerol were purchased from Merck Sdn. Bhd., Malaysia. Kenaf core fiber (KCF) with a particle size of 420 μm was bought from the National Kenaf and Tobacco Board, Malaysia. Dolomite (Dol) with a particle size of 177 μm was obtained from Dolomite Kangar Manufacturing Sdn. Bhd., Malaysia. All materials were used as received without further modification.

2.2. Preparation of Biocomposite Films

The biocomposite films were prepared by solution casting. First, 1.6 g of glycerol was poured into a 100 mL beaker, followed by KCF and 56 g of distilled water. The mixture was then stirred using a magnetic stirrer. 2.4 g of agar was slowly added to the mixture, then heated to 90°C and stirred at 1500 rpm until all the agar dissolved. The mixed solution was immediately cast into a 14 cm internal-diameter glass Petri dish and allowed to cool to room temperature (25°C) for gelation. The gel was then slowly dried in an oven at 40°C for 48 hours to obtain a freestanding biocomposite film. The content of KCF was varied from 4 to 16 wt.% relative to

the weight of agar, while the weight ratio of agar to glycerol was fixed at 3:2. The agar film without KCF (0 wt.%) was also prepared for comparison purposes. Additionally, the biocomposite films incorporating Dol were prepared using the same procedure, with the Dol content ranging from 1 to 4 wt.% relative to the weight of agar/KCF, while the KCF content was fixed at 4 wt.%.

2.3. Tensile Test of Biocomposite Films

The biocomposite films were cut into a rectangular shape using a die cutter for tensile testing. The tensile strength, elongation at break, and Young's modulus were determined according to ASTM D638-22 using a universal testing machine (Instron, model 5567) equipped with a 30 kN load cell. The crosshead speed and the gauge length were 5 mm min^{-1} and 30 mm, respectively. Nine biocomposite films from each percentage were tested to obtain the mean values. The tensile test results were analyzed using statistical software (Microsoft® Excel®). One-way analysis of variance (ANOVA) was used to determine whether KCF or Dol content had a significant effect on the tensile strength, elongation at break, and Young's modulus of the films at the 95% confidence level.

3. RESULTS AND DISCUSSION

3.1. Tensile Properties of Agar/KCF Biocomposite Films

Figure 2 shows the tensile strength of the agar/KCF biocomposite films at different KCF contents. It can be observed that the biocomposite films exhibited an increase in tensile strength from 12.52 MPa without KCF to 14.85 MPa at a KCF content of 4 wt.%. The tensile strength remained at 8 wt.% KCF, reaching 14.72 MPa, before decreasing to 12.23 and 11.74 MPa at 12 and 16 wt.% KCF, respectively. The highest tensile strength was obtained at 4 wt.% KCF, suggesting that this content provided effective reinforcement within the agar matrix. At low KCF content, fiber dispersion and interfacial interaction may facilitate efficient stress transfer between the agar matrix and KCF. Conversely, the decrease in tensile strength at higher KCF contents may be associated

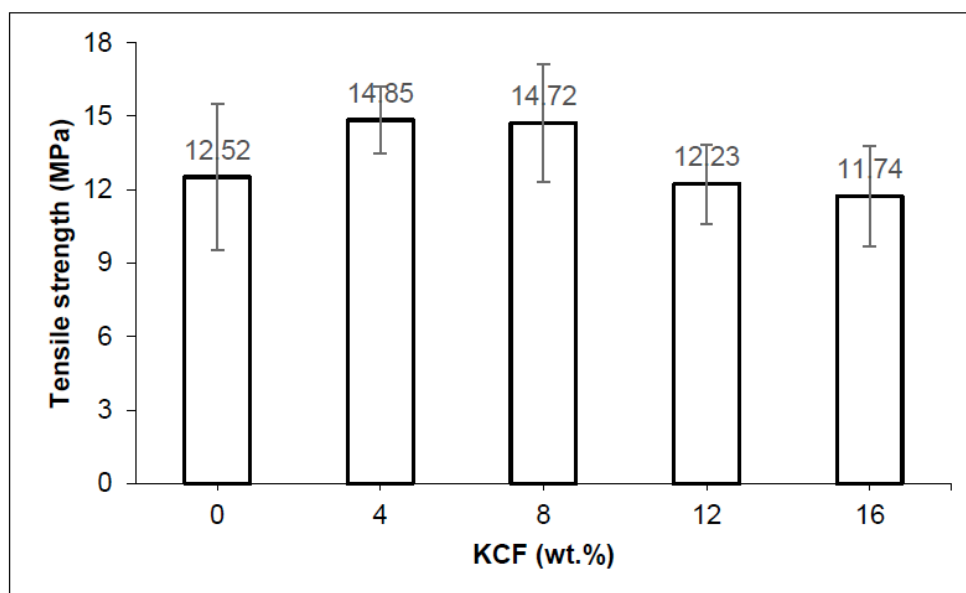


Figure 2: Tensile strength of the agar/KCF biocomposite films at different KCF contents.

with possible fiber agglomeration [12] and less effective matrix-fiber interfacial adhesion, which could contribute to stress concentration and the formation of weaker regions within the films. Therefore, these results indicate that the incorporation of KCF improves tensile strength at an appropriate content.

Figure 3 shows the elongation at break of the agar/KCF biocomposite films at different KCF contents. It can be seen that the biocomposite films exhibited a decrease in elongation at break with increasing KCF content. The film without KCF recorded the highest elongation at break of 15.23 mm, which decreased to 12.14 mm at 4 wt.% KCF and 9.34 mm at 8 wt.% KCF. Further increases in KCF content to 12 and 16 wt.% reduced the elongation at break to 6.92 and 6.52 mm,

respectively. This decreasing trend indicates that increasing KCF content restricted the flexibility and deformation of the agar-based biocomposite films. The lignocellulosic fibers may limit the mobility of agar polymer chains and reduce their ability to extend under loading. Higher fiber contents may also restrict polymer chain mobility, reduce matrix-fiber interactions, constrain matrix deformation, and consequently produce more rigid biocomposite structures [13]. Thus, the elongation at break results reveal that the presence of KCF decreases film extensibility.

Figure 4 shows the Young's modulus of the agar/KCF biocomposite films at different KCF contents. It can be noticed that the biocomposite films exhibited an increase in Young's modulus with increasing KCF

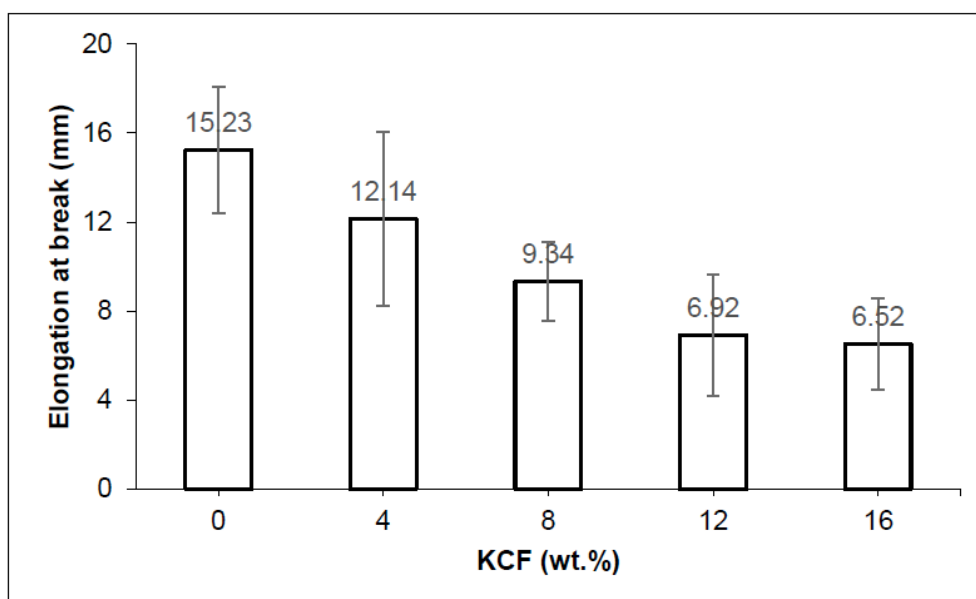


Figure 3: Elongation at break of the agar/KCF biocomposite films at different KCF contents.

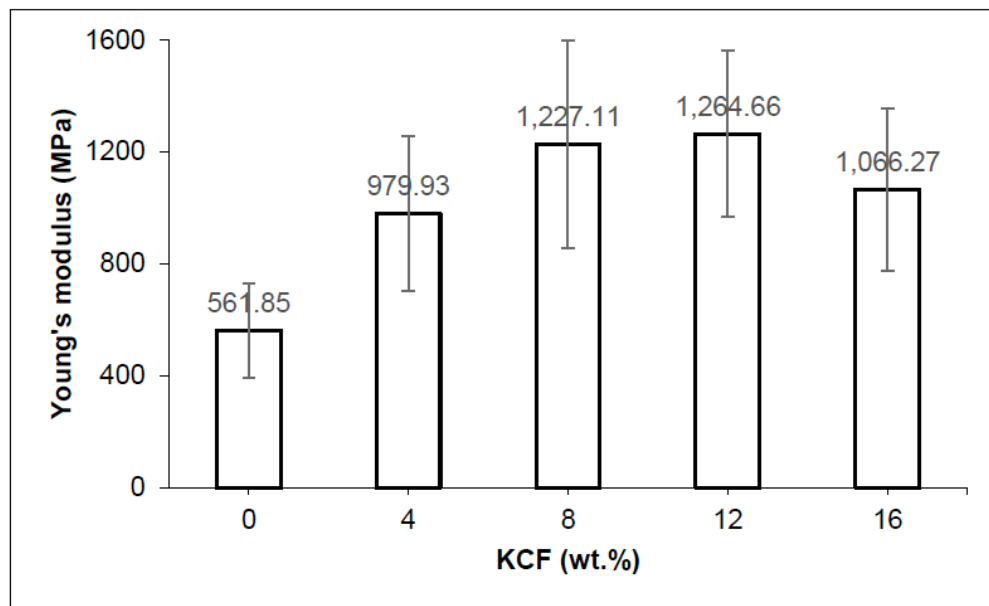


Figure 4: Young's modulus of the agar/KCF biocomposite films at different KCF contents.

content up to 12 wt.%. The film without KCF recorded the lowest Young's modulus of 561.85 MPa, which increased to 979.93 MPa at 4 wt.% KCF and 1,227.11 MPa at 8 wt.% KCF. The highest Young's modulus of 1,264.66 MPa was obtained at a KCF content of 12 wt.%, indicating greater film stiffness. This increase may be attributed to the polar nature of KCF and its reinforcing effect within the agar matrix [2], which restricts polymer chain mobility and deformation under applied stress; however, at 16 wt.% KCF, Young's modulus decreased to 1,066.27 MPa, possibly because excessive fiber content reduced reinforcement through poorer fiber dispersion. Hence, the Young's modulus results imply that the incorporation of KCF increases overall film stiffness.

Tables 1 to 3 present the one-way ANOVA results for the tensile strength, elongation at break, and

Young's modulus of agar/KCF biocomposite films at different KCF contents. Five biocomposite percentages were evaluated, with nine replicates tested for each percentage, giving 45 observations for each tensile property. The sources of variation were divided into between groups (BG) and within groups (WG) categories. The F-value represents the ratio of the BG mean square to the WG mean square. The F-values obtained for tensile strength, elongation at break, and Young's modulus were 4.09754, 15.81212, and 8.51456, respectively. Their corresponding P-values were 0.00709, 7.69×10^{-8} , and 4.55×10^{-5} , respectively. Since all P-values were below 0.05, the null hypothesis was rejected [1]. Therefore, KCF content had statistically significant effects on all three tensile properties of the agar/KCF biocomposite films at the 95% confidence level.

Table 1: One-way ANOVA Results of the Tensile Strength of the Agar/KCF Biocomposite Films at Different KCF Contents

Source of Variation	SS	df	MS	F-value	P-Value
BG	76.99188	4	19.24797	4.09754	0.00709
WG	187.89803	40	4.69745	-	-

SS = sum of square, df = degree of freedom, and MS = mean square.

Table 2: One-way ANOVA Results of the Elongation at Break of the Agar/KCF Biocomposite Films at Different KCF Contents

Source of Variation	SS	df	MS	F-value	P-Value
BG	485.82011	4	121.45503	15.81212	7.69×10^{-8}
WG	307.24533	40	7.68113	-	-

SS = sum of square, df = degree of freedom, and MS = mean square.

Table 3: One-way ANOVA Results of the Young's Modulus of the Agar/KCF Biocomposite Films at Different KCF Contents

Source of Variation	SS	df	MS	F-value	P-Value
BG	2847656	4	711914	8.51456	4.55×10^{-5}
WG	3344455	40	83611	-	-

SS = sum of square, df = degree of freedom, and MS = mean square.

Tukey's HSD post-hoc test at the 95% confidence level ($\alpha = 0.05$) was conducted to clarify which contents account for the significant overall ANOVA results. For tensile strength, the ANOVA showed a significant overall effect of KCF content (ANOVA, $p = 0.00709$). Tukey's HSD showed that 4 and 8 wt.% KCF had significantly higher tensile strengths than 16 wt.% KCF ($p = 0.0322$ and 0.0431 , respectively). No other pairwise differences were significant. For elongation at break (ANOVA, $p = 7.69 \times 10^{-8}$), significant differences were found between 0 and 8 wt.% ($p = 0.0005$), 0 and 12 wt.% ($p < 0.0001$), 0 and 16 wt.% ($p < 0.0001$), 4 and 12 wt.% ($p = 0.0024$), and 4 and 16 wt.% KCF ($p = 0.0010$). For Young's modulus (ANOVA, $p = 4.55 \times 10^{-5}$), the film without KCF differed significantly from 4 wt.% ($p = 0.0299$), 8 wt.% ($p = 0.0002$), 12 wt.% ($p = 0.0001$), and 16 wt.% KCF ($p = 0.0055$). No significant differences were observed among the KCF-incorporating films.

3.2. Tensile Properties of Agar/KCF/Dol Biocomposite Films

Although the highest Young's modulus was obtained at 12 wt.% KCF, 4 wt.% KCF was selected for the subsequent dolomite study because it exhibited the highest tensile strength of 14.85 MPa. Since tensile strength represents the ability of the biocomposite film

to withstand applied stress before failure, it was considered the primary criterion for selecting the KCF content. Furthermore, the addition of 4 wt.% KCF increased the Young's modulus compared with the agar film without KCF, while retaining a higher elongation at break than films incorporating higher KCF contents. Therefore, 4 wt.% KCF was considered a suitable content for investigating the subsequent effects of dolomite incorporation.

Figure 5 shows the tensile strength of the agar/KCF/Dol biocomposite films at different Dol contents. It can be observed that the biocomposite films exhibited a slight decrease in tensile strength after incorporating 1 and 2 wt.% Dol. The tensile strength decreased from 14.85 MPa without Dol to 13.95 MPa at 1 wt.% Dol and reached the lowest value of 13.30 MPa at 2 wt.% Dol. However, further incorporation of Dol increased the tensile strength to 14.37 MPa at 3 wt.% and 15.72 MPa at 4 wt.% Dol. The highest tensile strength was therefore obtained at a Dol content of 4 wt.%, exceeding that of the film without Dol. This improvement may be attributed to the abrasive nature of Dol, which provides mechanical interlocking [8] and facilitates effective stress transfer throughout the biocomposite structure. Lower Dol contents provided insufficient reinforcement to enhance the tensile performance. Therefore, these results indicate that the

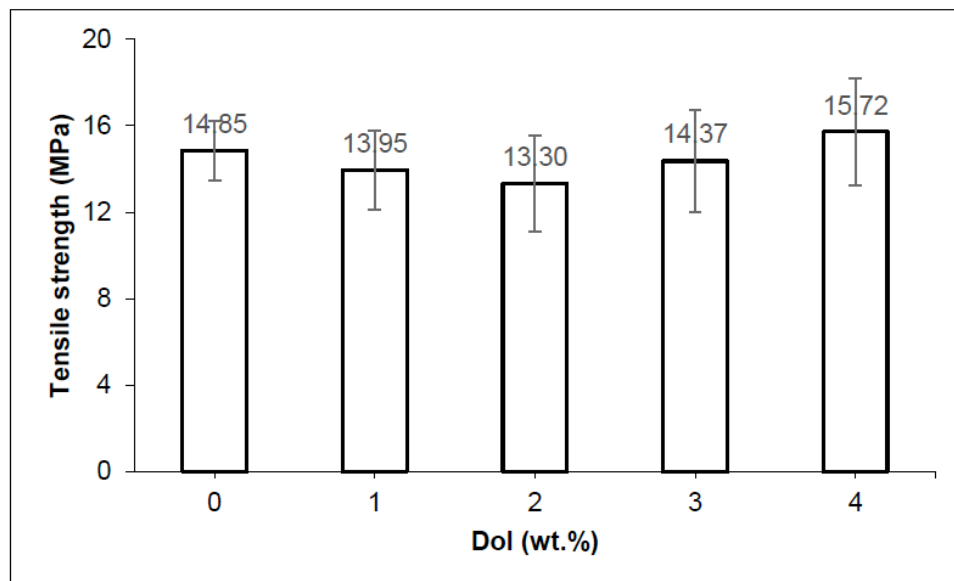


Figure 5: Tensile strength of the agar/KCF/Dol biocomposite films at different Dol contents.

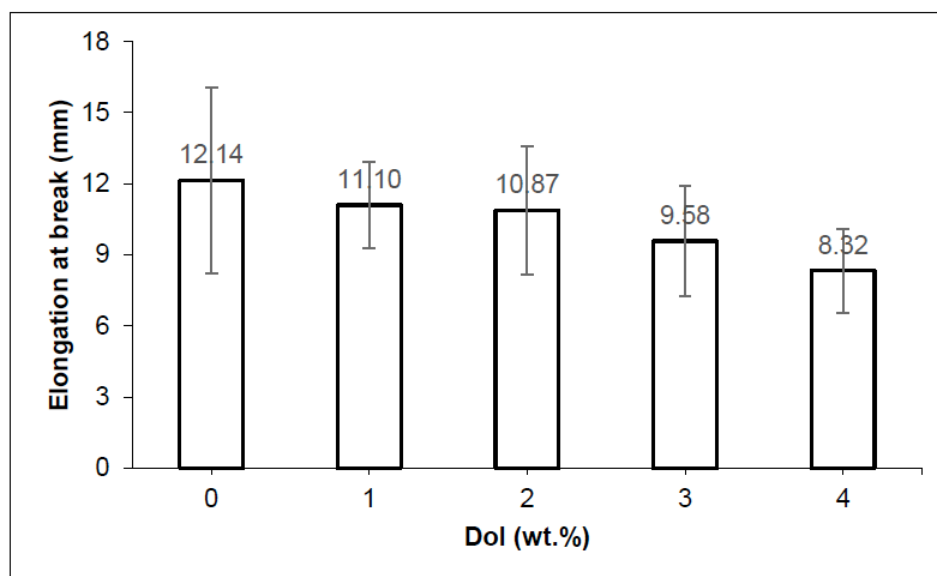


Figure 6: Elongation at break of the agar/KCF/Dol biocomposite films at different Dol contents.

incorporation of Dol improves tensile strength at higher contents.

Figure 6 shows the elongation at break of the agar/KCF/Dol biocomposite films at different Dol contents. It can be seen that the biocomposite films exhibited a gradual decrease in elongation at break with increasing Dol content. The film without Dol recorded the highest elongation at break of 12.14 mm, which decreased to 11.10 mm at 1 wt.% Dol and 10.87 mm at 2 wt.% Dol. Further increases in Dol content to 3 and 4 wt.% continuously reduced the elongation at break to 9.58 and 8.32 mm, respectively. This decreasing trend indicates that Dol incorporation progressively reduced the extensibility and flexibility of the biocomposite films. The dispersed Dol particles may restrict the mobility of agar polymer chains and

limit deformation of the matrix under applied loading [11]. Increasing Dol content results in a more rigid biocomposite structure with reduced ability to elongate before failure. Thus, the elongation at break results suggest that the presence of Dol reduces film extensibility.

Figure 7 shows the Young's modulus of the agar/KCF/Dol biocomposite films at different Dol contents. It can be noticed that the biocomposite films initially exhibited a decrease in Young's modulus from 979.93 MPa without Dol to 867.47 MPa at 1 wt.% Dol. Subsequently, Young's modulus increased to 946.30 MPa at 2 wt.% Dol and 1,144.17 MPa at 3 wt.% Dol. The highest Young's modulus of 1,531.80 MPa was obtained at a Dol content of 4 wt.%, indicating that this percentage produced the stiffest biocomposite film.

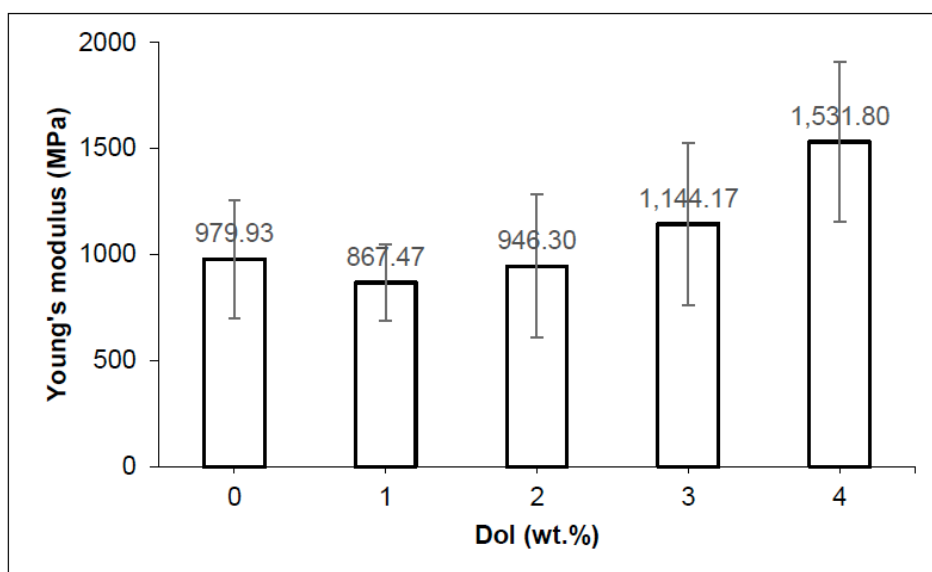


Figure 7: Young's modulus of the agar/KCF/Dol biocomposite films at different Dol contents.

The increase at higher Dol contents may be due to the rigid mineral particles restricting the mobility and deformation of the agar matrix. Sufficient Dol particles may also improve load transfer within the biocomposite structure [14], thereby increasing resistance to elastic deformation. The lower modulus at 1 wt.% suggests insufficient reinforcement at low Dol content. Hence, the Young's modulus results signify that the incorporation of Dol increases film stiffness at higher contents.

Tables 4 to 6 present the one-way ANOVA results for the tensile strength, elongation at break, and Young's modulus of agar/KCF/Dol biocomposite films at different Dol contents. Five biocomposite percentages were evaluated, with nine replicates tested for each percentage, giving 45 observations for each tensile property. The sources of variation were divided into between groups (BG) and within groups (WG) categories. The F-value represents the ratio of the BG mean square to the WG mean square. The F-values for tensile strength, elongation at break, and Young's modulus were 1.71412, 2.81816, and 6.16695, respectively, with corresponding P-values of 0.16589, 0.03764, and 0.00058. Therefore, the null hypothesis was retained for tensile strength but rejected for elongation at break and Young's modulus. Consequently, Dol content had a significant effect on elongation at break and Young's modulus, but did not significantly affect tensile strength, at the 95% confidence level.

Tukey's HSD post-hoc test at the 95% confidence level ($\alpha = 0.05$) was also conducted to clarify which contents account for the significant overall ANOVA results. For tensile strength, the overall ANOVA was not significant (ANOVA, $p = 0.16589$); therefore, the differences among Dol contents were not statistically significant, and post-hoc interpretation is unnecessary. For elongation at break (ANOVA, $p = 0.03764$), Tukey's HSD identified only 0 versus 4 wt.% Dol as significantly different ($p = 0.0298$). The other pairwise comparisons were not significant. For Young's modulus (ANOVA, $p = 0.00058$), 4 wt.% Dol showed significantly higher values than 0 wt.% ($p = 0.0062$), 1 wt.% ($p = 0.0007$), and 2 wt.% Dol ($p = 0.0033$). The difference between 3 and 4 wt.% Dol was not statistically significant ($p = 0.0953$).

4. CONCLUSIONS

The effects of KCF and Dol contents on the tensile properties of agar-based biocomposite films were investigated in this study. Incorporating KCF increased tensile strength from 12.52 MPa to a maximum of 14.85 MPa at 4 wt.% KCF, while Young's modulus increased considerably, reaching 1,264.66 MPa at 12 wt.% KCF. Conversely, elongation at break continuously decreased with increasing KCF content. One-way ANOVA confirmed that KCF content significantly affected tensile strength, elongation at break, and Young's modulus. Based on these results, 4

Table 4: One-way ANOVA Results of the Tensile Strength of the Agar/KCF/Dol Biocomposite Films at Different Dol Contents

Source of Variation	SS	df	MS	F-value	P-Value
BG	30.02272	4	7.50568	1.71412	0.16589
WG	175.14928	40	4.37873	-	-

SS = sum of square, df = degree of freedom, and MS = mean square.

Table 5: One-way ANOVA Results of the Elongation at Break of the Agar/KCF/Dol Biocomposite Films at Different Dol Contents

Source of Variation	SS	df	MS	F-value	P-Value
BG	78.49863	4	19.62466	2.81816	0.03764
WG	278.54555	40	6.96364	-	-

SS = sum of square, df = degree of freedom, and MS = mean square.

Table 6: One-way ANOVA Results of the Young's Modulus of the Agar/KCF/Dol Biocomposite Films at Different Dol Contents

Source of Variation	SS	df	MS	F-value	P-Value
BG	2523001	4	630750	6.16695	0.00058
WG	4091167	40	102279	-	-

SS = sum of square, df = degree of freedom, and MS = mean square.

wt.% KCF was selected for subsequent Dol incorporation. Incorporating Dol produced the highest tensile strength and Young's modulus of 15.72 and 1,531.80 MPa, respectively, at a Dol content of 4 wt.%, whereas elongation at break progressively decreased. One-way ANOVA showed that Dol content significantly affected elongation at break and Young's modulus but not tensile strength. In conclusion, incorporating KCF and Dol enhanced the stiffness of agar-based biocomposite films while reducing their extensibility and improving overall tensile performance.

ACKNOWLEDGEMENTS

This initial study was funded by Universiti Putra Malaysia through the Grant Putra Initiative Scheme (project number: GPI/2025/9839400). The authors thank Dolomite Kangar Manufacturing Sdn. Bhd., Malaysia, for providing dolomite and technical knowledge. The first author also thanks his family and the INTROP staff for supporting him in conducting this study even though he was fighting stage 3 cancer.

AUTHOR'S CONTRIBUTION STATEMENT

A.A.S. conceived and designed the study. N.F.N.M.A. performed the experiments and collected the data. A.A.S. conducted numerical simulations and formal analysis. A.A.S. wrote the original draft, and M.A.Z. contributed to reviewing and editing the manuscript. A.A.S. supervised the project and acquired funding.

CONFLICTS OF INTEREST

The authors declare no conflicts of interest.

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