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Development of Eco-Friendly Biocomposite Films Based on Whey Protein Isolate and Carboxymethyl Cellulose with Silica as a Filler

Mukhlishien*, **Azwar**

Chemical Engineering Department, Engineering Faculty, University Syiah Kuala, Banda Aceh
***Corresponding author**: mukhlishien@usk.ac.id

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Abstract
This research endeavor focuses on the development of biodegradable composite films. The constituents utilized comprise Whey Protein Isolate (WPI) and Carboxymethyl Cellulose (CMC). Additionally, varying quantities of silica filler are incorporated into the formulations. Films were created using two different CMC amounts (0.75g and 1g) and varying silica levels (0g, 0.05g, 0.15g, 0.25g, 0.35g, and 0.45g). The research systematically examined how the addition of silica affected the films' thickness (which increased from 0.126 mm to 0.371 mm with higher silica content), water absorption, biodegradability, and surface structure. Silica addition also reduced water absorption significantly, improving the film's resistance to swelling. Biodegradation tests showed all samples met the SNI biodegradability standard (>60%), although higher silica levels reduced the rate of degradation due to its hydrophobic nature. SEM analysis illustrated that films with added silica exhibited smoother, denser surfaces with fewer voids, indicating improved particle distribution and better structural integrity. Overall, the incorporation of silica and CMC successfully enhanced the functional performance of WPI-based biocomposite films. The optimal silica concentration (0.05–0.25 g) provided a good balance between mechanical strength, water resistance, and environmental degradability.
Keywords: *biocomposite film, whey protein isolate (wpi), carboxyl methyl cellulose (cmc), silica filler, biodegradable packaging*

Abstrak
Penelitian ini berfokus pada pengembangan film komposit biodegradabel. Bahan-bahan yang digunakan meliputi *Whey Protein Isolate* (WPI) dan *Carboxymethyl Cellulose* (CMC). Selain itu, berbagai jumlah pengisi silika dimasukkan ke dalam formulasi. Film-film biokomposit dibuat menggunakan dua jumlah CMC yang berbeda (0,75 gram dan 1 gram), dengan silika ditambahkan pada konsentrasi 0, 0,05, 0,15, 0,25, 0,35, dan 0,45 gram. Penelitian ini secara sistematis menguji pengaruh silika terhadap ketebalan film (yang meningkat dari 0,126 mm menjadi 0,371 mm seiring dengan peningkatan kandungan silika), penyerapan air, biodegradabilitas, dan morfologi permukaan. Penambahan silika juga menurunkan daya serap air secara signifikan, sehingga meningkatkan ketahanan film terhadap pembengkakan. Uji biodegradasi menunjukkan bahwa semua sampel memenuhi standar biodegradabilitas SNI (>60%), meskipun tingkat degradasi menurun seiring bertambahnya kadar silika karena sifat hidrofobiknya. Analisis SEM memperlihatkan bahwa film yang mengandung silika memiliki permukaan yang lebih halus, padat, dan minim pori, yang mengindikasikan distribusi partikel yang lebih merata dan struktur yang lebih kuat. Secara keseluruhan, penambahan silika dan CMC berhasil meningkatkan performa fungsional dari film biokomposit berbasis WPI. Konsentrasi silika yang optimal (0,05–0,25 g) memberikan keseimbangan yang baik antara kekuatan mekanik, ketahanan terhadap air, dan kemampuan terurai di lingkungan.
Kata Kunci: *film biokomposit, isolat protein whey (wpi), karboksil metil selulosa (cmc), filler silika, kemasan biodegradable*

1. Introduction

The utilization of biodegradable materials in the realm of food packaging is experiencing a significant escalation. This phenomenon is primarily propelled by the imperative to preserve product integrity. Moreover, the extension of shelf life constitutes an additional rationale for this trend. This surge also endeavors to enhance environmental sustainability. Bio-based polymers, including polysaccharides, lipids, and proteins, are increasingly recognized as viable alternatives to petroleum-derived plastics. This transition responds to the escalating demand for materials that are environmentally benign. Furthermore, the employment of renewable natural resources represents an additional benefit. At present, a variety of biopolymers are actively utilized and commercially manufactured as thin films within the food packaging

sector. Illustrative examples encompass polylactic acid (PLA), starch, chitosan, cellulose, and proteins [1], [2].

Whey protein isolate (WPI) is a biologically derived compound known for its high protein content. It is extracted from dairy by-products generated during the production of cheese and tofu. Previous studies have highlighted WPI's excellent film-forming and gas barrier properties, making it superior to many petroleum-based polymers in this regard [3]. However, WPI-based films often fall short in packaging applications due to their poor moisture resistance and weak mechanical strength—limitations largely attributed to the high presence of hydrophilic amino acids [4]. This study explores the incorporation of nanoscale additives into WPI film structures to enhance or chemically modify their performance and address these inherent weaknesses. To resolve these issues, carboxyl methyl cellulose (CMC) was introduced into the formulation. CMC served as a plasticizer in the development of the biocomposite films, chosen for its affordability and wide availability.

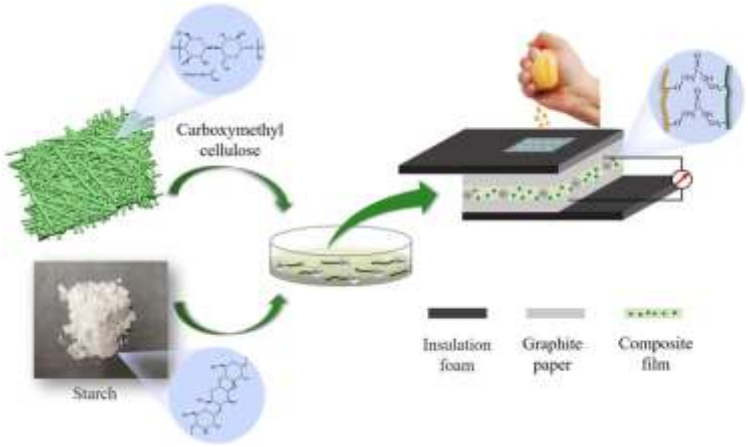

Fig. 1: Schematic representation of the starch/CMC composite film structure used for the rapid detection of juice quality [5]

Carboxymethyl Cellulose (CMC) is a derivative of cellulose synthesized via an esterification process. This biopolymer exhibits a wide array of applications, frequently utilized as a stabilizing agent, a thickening agent within the alimentary sector, and as a crucial constituent in the fabrication of bioplastics, which typically entails the incorporation of crosslinking agents to augment their mechanical characteristics. Investigations conducted by Hasanah and associates reveal that the incorporation of CMC substantially influences the biodegradation kinetics of bioplastics, wherein an increased concentration of CMC added is positively associated with an accelerated rate of film disintegration by microbial organisms. However, a significant obstacle faced is the proclivity of biocomposite films to dissolve upon exposure to aqueous environments, which is ascribed to the presence of starch molecules that tend to retain water, consequently impeding the overall solubility of the starch component. Conversely, the study by Bayu and their team reveals that the incorporation of CMC into the biocomposite film formulation results in a smoother film surface structure. To further enhance the characteristics of these biocomposite films, fillers are often added, such as silica, which is known to possess naturally readily degradable properties [6].

Silica is an effective filler for biocomposite films due to its well-known porous structure, significant surface activity, and compatibility with biological systems. The mechanical properties of corn starch and LDPE biocomposites are enhanced through the integration of silica into their matrix. The increase in filler concentration can integrate the biocomposite film more effectively. As a result, increasing silica content can influence the physical, mechanical, and thermal characteristics of the WPI/CMC-based biocomposite films, thanks to the beneficial and functional properties of silica [7].

Consequently, this research endeavor concerning biocomposite films seeks to investigate the parameters of thickness, water absorption capacity, physical characteristics, mechanical attributes, and environmental degradability. The assessment of physical characteristics was conducted utilizing Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM). Furthermore, the evaluation of mechanical attributes encompassed the determination of tensile strength and elongation properties.

2. Material and Methods

2.1. Material

The materials used in this study were CMC powder as a plasticizer, WPI as the main biopolymer, silica as a filler, and distilled water as the solvent. Each component was selected to enhance the film's mechanical and biodegradable properties.

2.2. Methods

2.2.1. Dissolution Stage of WPI and CMC

The preparation began by dissolving 5 grams of WPI in 100 mL of distilled water. This mixture was continuously stirred for approximately 1.5 hours to ensure homogeneity. Once fully dissolved, the solution was heated to 70°C and maintained at that temperature for 1 hour. After heating, CMC was added in two different concentrations: 0.75 grams and 1 gram. The solution was then allowed to cool naturally to room temperature before proceeding to the next stage.

2.2.2. Silica Dissolution Stage with Distilled Water

Silica was mixed into 50 mL of distilled water and treated using an ultrasonic bath for 1 hour at room temperature. Five different concentrations were used: 0.05, 0.15, 0.25, 0.35, and 0.45 grams. This ultrasonic treatment helped break down particles for better dispersion. The resulting mixture was then heated at 70°C for 30 minutes. This step ensured improved homogeneity and stability of the silica solution.

2.2.3. Biocomposite Film Preparation Stage

At this stage, the WPI/CMC mixture was combined with the silica solution to form a homogeneous blend. The resulting mixture was poured into petri dishes to shape the films. The films were then dried at 55°C to allow proper solvent evaporation. Once dried, they were cooled at room temperature. This process resulted in the formation of uniform biocomposite films.

2.2.4. Biocomposite Film Analysis

The synthesized biocomposite films underwent a comprehensive series of evaluations, encompassing thickness, swelling, tensile strength, elongation, and degradation assessments to ascertain their physical characteristics and functional efficacy. The objective of the thickness evaluation was to ascertain the consistency of the films produced. In the execution of this assessment, the films were sectioned into 2 x 2 cm fragments, and the thickness was quantified utilizing a digital caliper with a precision of 0.01 mm. To guarantee the reliability of the findings, the thickness was measured at five distinct locations across each specimen, and the mean of these measurements was computed [8]. Swelling assessments were conducted by segmenting a 50 mm length of the biocomposite film and recording its initial weight. Subsequent to the cleansing of the film with tissue to eliminate any residual moisture, the film was reweighed to evaluate the degree of swelling induced by water absorption.

The material's ability to retain water is determined by this test, which is important for applications where moisture resistance is required. Conducted according to ASTM D-682 standards, the tensile strength test is essential for understanding the maximum stress the biocomposite material can withstand before failure. The tests were performed using a Computer Universal Testing Machine HT-2402, which automatically calculates the maximum load value during the test. Elongation, the change in length (strain) of the material under applied stress, was measured in parallel with the tensile strength tests. Each biocomposite film was cut into 2 x 2 cm pieces. In this study, degradation testing was performed using the soil burial method. This method involves burying the biocomposite film samples in soil. The samples were weighed before burial and then weighed again at specific time intervals during the burial period to monitor the degradation process [9].

The morphological structure of the biocomposite films was examined using Scanning Electron Microscopy (SEM). Each sample was cut into 2 x 2 cm pieces for SEM analysis. The SEM results revealed the distribution of filler particles within the matrix, helping to determine whether the filler particles were evenly dispersed throughout the matrix.

Functional group analysis of the biocomposite films was performed using a Shimadzu FTIR spectrometer model IR Prestige 21. The purpose of this test was to identify the functional groups present in the biocomposite films. For this analysis, the samples were placed in the specimen holder and positioned in the spectrometer chamber. The spectral range was measured between 4500-500 cm^{-1} with a personal computer controlling the spectrometer operation [10]. This analysis helps to understand the chemical composition and interactions within the biocomposite material.

3. Results and Discussion

3.1 Thickness of Biocomposite Film

Depicted in **Figure 2** are the thickness evaluations obtained for a sequence of biocomposite films. These films were meticulously designed employing Whey Protein Isolate (WPI) as the principal matrix material, in conjunction with two discrete concentrations of Carboxymethyl Cellulose (CMC), specifically 0.75 grams and 1 gram. To enhance the understanding of the impact of filler content, diverse amounts of silica particles were incorporated into the film formulations. The specific concentrations used ranged from 0, 0.05, 0.15, 0.25, 0.35, to 0.45 grams.

An analytical review of the data visually depicted in **Figure 2** discloses a definitive correlation between the presence and concentration of silica filler and the consequent thickness of the biocomposite films. It is apparent that the biocomposite films that incorporate silica demonstrate a spectrum of thickness values. This range extends from a minimum of 0.126 mm to a maximum of 0.371 mm. This observed spectrum accentuates the impact of the integrated silica on the physical dimensions of the films.

Moreover, the findings uniformly demonstrate a favorable correlation between the concentration of silica and the thickness of the film across all examined samples. As the amount of silica introduced into the biocomposite formulation is incrementally increased, a corresponding and proportional increase in the measured thickness of the resultant film is observed. This trend strongly suggests that the inclusion of silica particles plays a substantial and direct role in determining the final thickness of the biocomposite material. These particles act as a filler material within the film-forming process.

The observed increase in film thickness with higher silica concentrations can be attributed to the physical accumulation of these inorganic particles within the continuous biopolymeric matrix. The presence of these dispersed silica particles effectively increases the solid volume of the film, thereby leading to a greater overall thickness. This incorporation of filler not only contributes to the bulk of the material but can also influence other structural properties of the film.

Similarly, Carboxymethyl Cellulose (CMC) acts as a binder within the biocomposite system. This role also contributes to the overall film thickness. CMC, a polysaccharide known for its film-forming capabilities, interacts with the WPI matrix and the silica particles. The addition of CMC likely enhances the structural integrity of the film by facilitating a more cohesive and interconnected network of the constituent materials This interaction between the CMC and the silica particles appears to reinforce the film's structure. This reinforcement allows for a greater degree of control over the final thickness achieved. The combined effect of increased silica loading and the presence of CMC as a binding agent contributes to the observed variations in the thickness of the developed biocomposite films.

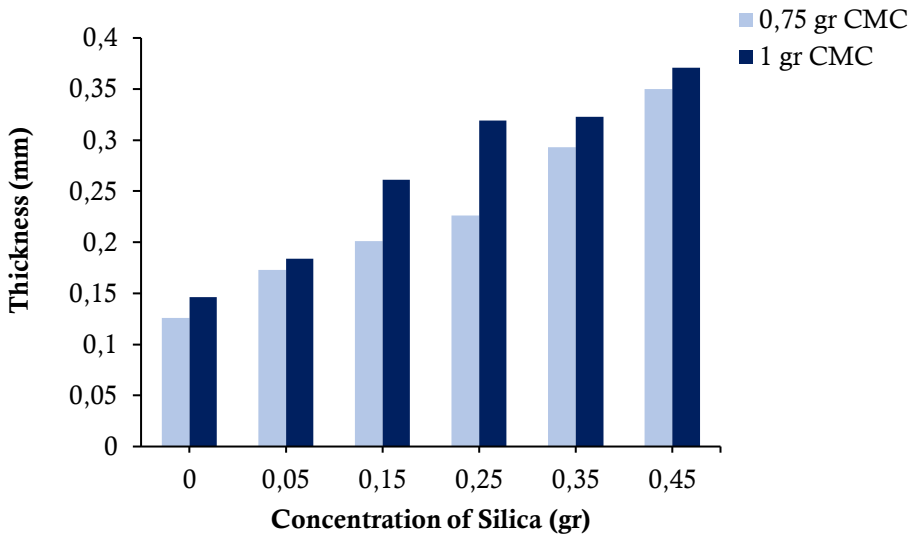

Fig. 2: The effect of silica concentration on the thickness of WPI/CMC biocomposite films

The biocomposite films developed in the present investigation conform to the established criteria. This is particularly true for those incorporating silica concentrations of 0.05 grams, 0.15 grams, and 0.25 grams. These criteria are promulgated by the food industry for biocomposite materials utilized in food packaging applications. The maximum thickness of the films generated in this inquiry was recorded at 0.25 mm, which adheres to the Japanese Industrial Standard (JIS) pertaining to biocomposite materials. This observation is noteworthy, as it suggests that these films possess considerable potential for application in

food packaging, wherein material thickness and structural integrity are paramount considerations for the safeguarding of food products.

The biocomposite films synthesized in the present investigation conform to the regulatory benchmarks. This is especially true for those incorporating silica additions of 0.05 grams, 0.15 grams, and 0.25 grams. These benchmarks are established by the food industry for biocomposite materials employed in food packaging applications. Furthermore, the maximum film thickness recorded in this research was 0.25 mm. This measurement adheres to the Japanese Industrial Standard (JIS) pertaining to biocomposite materials. This observation is of considerable importance, as it suggests that these films possess the potential for application in food packaging, wherein material thickness and structural integrity are critical factors for the safeguarding of food products.

The biocomposite films synthesized in this investigation meet the criteria established by the food industry. This is particularly true for films incorporating silica additions of 0.05 grams, 0.15 grams, and 0.25 grams. These criteria are for biocomposite materials utilized in food packaging applications. Additionally, the maximum film thickness attained in this research was 0.25 mm, which adheres to the Japanese Industrial Standard (JIS) for biocomposite materials. This observation is of considerable importance, as it suggests that these films possess the potential for application in food packaging, where the thickness of materials and their structural integrity are critical factors in safeguarding food products. This study's findings show that adding silica and carboxymethyl cellulose (CMC) to the biocomposite formulation leads to a controlled increase in film thickness. This regulated enhancement makes these materials appropriate for food packaging applications. These applications align with established industry standards.

3.2 Water Absorption (Swelling) of Biocomposite Films

The swelling assessment was performed through the quantification of the mass of the biocomposite film coupled with an analysis of the water absorption kinetics. This methodology facilitated the evaluation of the quantity of water molecules assimilated by the biocomposite film mass. The outcomes from the water absorption examination of biocomposite films incorporated with silica are delineated in **Figure 3**. As illustrated in **Figure 3**, the incorporation of silica into the film specimens led to a diminishment in the percentage of water absorption. This phenomenon can be explicated by the role of silica as a filler, which is instrumental in enhancing the mechanical attributes and tensile strength of the biocomposite film. In the case of biocomposite films lacking silica, the utilization of 0.75 and 1 gram of Carboxymethyl Cellulose (CMC) as the plasticizing agent yielded water absorption percentages of 83.23% and 79.23%, correspondingly.

Upon the introduction of silica at various concentrations (0.05, 0.15, 0.25, 0.35, and 0.45 grams), the water absorption percentages for films with 0.75 grams of CMC were 79.61%, 75.48%, 73.29%, 68.56%, and 63.33%, respectively. In contrast, for films containing 1 gram of CMC, different water absorption percentages were recorded for the same silica concentrations. These percentages were 73.84%, 71.21%, 68.95%, 66.72%, and 60.86%, respectively. This indicates a trend of decreasing water absorption with increasing silica content across both CMC concentrations.

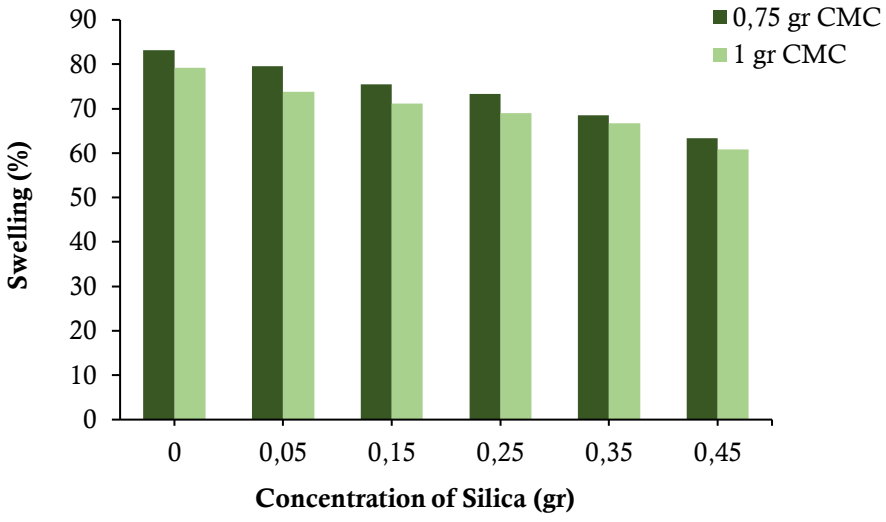

Fig. 3: Water absorption capacity of biocomposite films

The data collected clearly demonstrates an inverse relationship between the concentration of silica within the biocomposite film and its swelling capacity. Specifically, as the proportion of silica incorporated into the film matrix increases, a corresponding decrease in the measured swelling value of the material is observed. This suggests that the incorporation of silica reduces the film's capacity to absorb water. This trend suggests that lower water absorption values indicate improved properties of the film, while higher water absorption values may lead to film degradation. The addition of silica, being a filler and reinforcement material, helps reduce the porosity of the biocomposite film. When silica is integrated into the composite matrix, it occupies the voids between particulate matter or fibrous structures. This occupation results in an enhancement of the material's density and rigidity. Consequently, the interstitial spaces within the matrix that would ordinarily accommodate water are diminished, thus constraining the material's capacity for water absorption. The combination of the filler effect, specifically from the silica particles, plays a significant role in reducing the swelling behavior of the biocomposite film. This reduction in swelling is a desirable characteristic for enhancing the stability and durability of the film, particularly in applications where water resistance is crucial. Consequently, the integration of silica enhances the comprehensive efficacy of the biocomposite films, rendering them more appropriate for utilization in contexts such as food packaging and other environments that are sensitive to moisture [11].

3.3 Biodegradation Capability of Biocomposite Films.

The biodegradation assessment began with measuring the biocomposite film's dry mass before soil exposure. After the film was placed in the soil environment, its mass was measured again. The biodegradation percentage was calculated by dividing the film's mass after planting by its initial dry mass. This result was then multiplied by 100%. The aim of this assessment was to determine how quickly the bioplastic could naturally decompose. This decomposition process is driven by the microbial activity present in the soil. The findings of the biodegradation assessment for biocomposite films incorporating CMC and silica are illustrated in **Figure 4**. As depicted in **Figure 4**, the results of the biodegradation assessment reveal a range of biodegradation percentages across the various film samples.

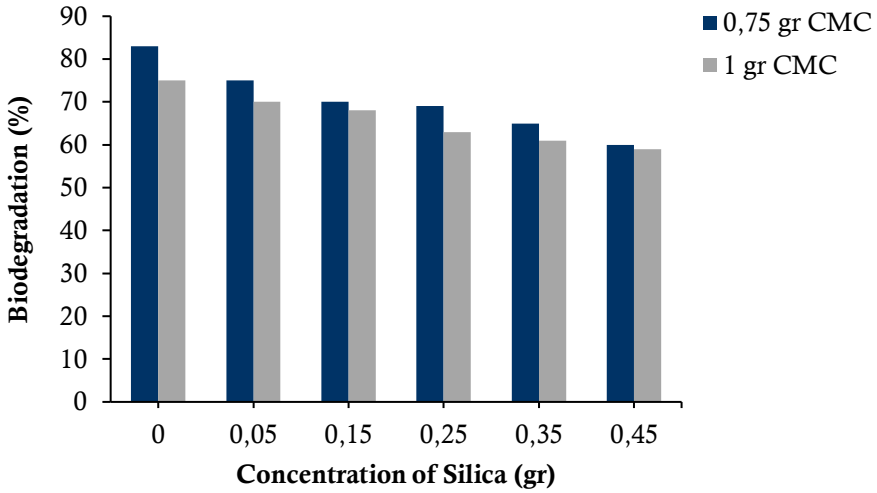

Fig. 4: The effect of silica addition on the biodegradation capability of biocomposite films

According to the Indonesian National Standard (SNI) 7188.7:2016, the required biodegradation threshold is a degradation value of >60%. The results shown in Figure 3 indicate that all biocomposite film samples met the standards set by the SNI. The highest biodegradation percentage was achieved by the sample with 0.75 grams of CMC without the addition of silica. Persentase degradasi maksimal, yang disebabkan oleh penambahan silika, tercatat pada film biokomposit yang terdiri dari 0,75 gram karboksimetil selulosa (CMC) dan 0,05 gram pengisi silika. Degradasi maksimal ini diatribusikan pada keberadaan silika dalam komposisi film tersebut. In contrast, the minimal degradation percentage was identified in the sample containing 1 gram of CMC alongside 0.45 grams of silica.

CMC, functioning as a plasticizer, possesses the capacity to retain moisture; consequently, an increase in the concentration of CMC correlates with a reduction in the biodegradation percentage of the biocomposite film [12]. Nevertheless, this attribute necessitates the reinforcement of fillers employed in the fabrication process of the biocomposite films. An elevation in silica concentration within the biocomposite films yields films characterized by diminished biodegradation capabilities, indicating an

enhancement in their resistance to degradation. Silica possesses hydrophobic properties. This characteristic inhibits water absorption from the surrounding soil environment, consequently constraining the degradation rate of the film As a result, the degradation potential of the biocomposite films is adversely affected as the concentration of silica within the film formulation escalates. This phenomenon can be attributed to the hydrophobic characteristics of silica, which impede the moisture content that is capable of permeating the film, thus obstructing the microbial activity essential for biodegradation [13]. These observations imply that while CMC augments the water retention characteristics of the film, consequently diminishing biodegradation, the silica filler further retards the degradation process owing to its hydrophobic nature. The interplay of these elements underscores the significance of both the plasticizer and the filler in influencing the environmental degradation potential of biocomposite materials.

3.4 Morphology of Biocomposite Films Using SEM

The findings derived from the Scanning Electron Microscopy (SEM) analysis are depicted in **Figure 5**. This figure elucidates the surface morphological characteristics of the WPI/CMC biocomposite films. Diverse concentrations of silica (0, 0.05, 0.15, 0.25, 0.35, and 0.45 grams) were integrated into these films. The presence of the biocomposite film is corroborated by the SEM analysis depicted in **Figure 5**. This figure delineates the variations in the surface morphology of the biocomposite films corresponding to the differing silica concentrations. In the instances of films devoid of silica, the samples containing 0.75 and 1 gram of CMC presented an irregular and porous surface morphology. In contrast, the biocomposite films incorporating silica concentrations of 0.05, 0.15, 0.25, 0.35, and 0.45 grams similarly exhibited irregular surface textures. However, these films showed a diminished occurrence of surface voids. This variation is attributed to the polymerization mechanism. Furthermore, the adhesion characteristics of silica particles to the surface of the biocomposite films also contribute to this difference. The irregular surface morphology of the biocomposite films can be elucidated by the non-homogeneous distribution of particles during the film formation process or potentially by the aggregation of particles throughout the fabrication procedure [14].

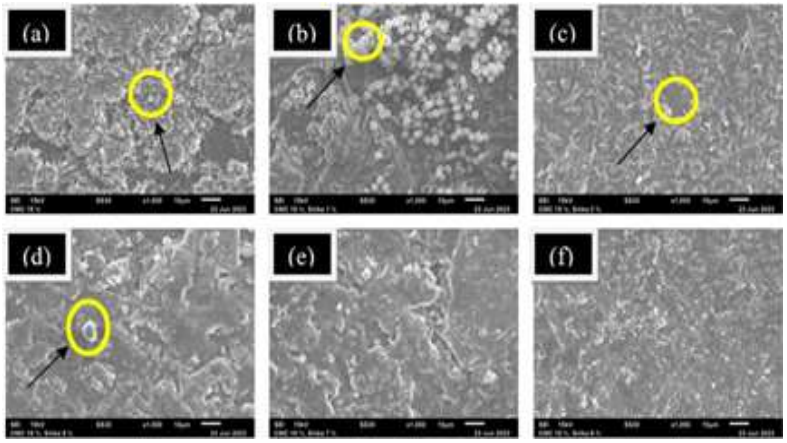

Fig. 5: SEM micrograph of the surface of WPI/CMC biocomposite films, CMC 0.75 g with varying silica concentrations.

The presence of silica particles is hypothesized to have significantly facilitated the diminution of surface voids within the biocomposite films. This phenomenon can be attributed to the function of silica as a filler material, which effectively occupies the interstices between polymer chains, consequently enhancing the overall structural integrity and morphology of the film. The results obtained from the SEM analysis further clarify that an elevation in silica concentration leads to a more densely packed configuration of the films, as evidenced by a decrease in the occurrence of pores and voids. This finding suggests that the addition of silica enhances the homogeneity of the film's surface. This may reinforce the mechanical properties of the material and its durability against environmental factors. The examination via scanning electron microscopy (SEM) substantiates a pivotal discovery. The incorporation of silica significantly influences the morphological properties of the biocomposite films. This influence is particularly evident in the reduction of surface porosity and the improvement of the material's structural integrity The discerned morphological trends bear significant implications for the practical utilization of these biocomposite films, especially in contexts where surface smoothness and material strength are paramount [15].

4. Conclusion

This study demonstrates a significant influence on the biocomposite films. This influence is due to both the addition of silica and the variation of CMC. The incorporation of silica as a filler enhances the film's thickness, mechanical integrity, and water resistance properties. An increasing concentration of silica resulted in thicker films due to particle accumulation within the polymer matrix. Furthermore, water absorption tests yielded important findings. The addition of silica effectively reduced the swelling behavior of the films, and higher silica concentrations resulted in significantly lower water uptake. This reduction is attributed to the decreased porosity and increased density of the film, which limits water penetration. The biodegradation analysis showed that all biocomposite films met the Indonesian National Standard (SNI) for biodegradability, with degradation percentages exceeding 60%. However, the rate of biodegradation was negatively affected by higher silica content. This is because silica's hydrophobic nature impedes microbial activity by limiting moisture absorption.

Thus, while silica strengthens the film, it also reduces its environmental degradability. SEM analysis supported these findings by revealing that silica enhances surface morphology, reducing surface voids and leading to more compact films. The uneven morphology observed in silica-free films improved with silica incorporation, suggesting better particle distribution and interfacial adhesion. In summary, the combination of whey protein isolate (WPI), carboxymethyl cellulose (CMC), and silica yields beneficial biocomposite films. These films exhibit enhanced structural characteristics and satisfactory biodegradability. These films exhibit promising potential for food packaging applications, particularly where mechanical strength and moderate biodegradability are desirable. Optimal silica content (0.05–0.25 g) offers the best balance between functional performance and environmental compatibility.

5. Acknowledgment

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