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A Preliminary Study on Biodegradability of Alkyl Ketene Dimer-Treated All-Cellulose Composites

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ABSTRACT

Cellulose is the most abundant natural organic polymer on Earth. It has been widely used to develop cellulose-based materials that can be tailored to exhibit tunable properties, including mechanical and physical properties, electrical conductivity, and other unique characteristics by incorporating additional functional components. Nowadays, a new class of monocomponent composites based on cellulosic materials, known as all-cellulose composites (ACCs), has attracted considerable interest among researchers. However, the hydrophilic nature of cellulose weakens its physical and mechanical properties due to the presence of numerous hydroxyl groups. Therefore, alkyl ketene dimer (AKD) was incorporated into the development of ACC to mitigate increased water absorption. Few studies have been reported on the physical and mechanical properties of AKD-treated ACC. However, no studies have addressed its biodegradation behavior. This study aims to investigate the role of AKD and the influence of temperature during soil burial testing on the biodegradation of AKD-treated ACC. The results indicated that AKD-treated ACC did not experience any degradation at either room temperature or 40°C, with negligible mass loss observed after 49 days of soil burial testing. In contrast, ACC exhibited surface discoloration and fiber loosening after burial. The mass loss for ACC was recorded as 4.6 and 12.2% at room temperature and 40°C, respectively. These findings suggest that the hydrophobic nature of AKD may impede ACC biodegradation. This study also relates to the Sustainable Development Goals (SDGs), including SDGs 6, 9, and 12-15.

Keywords: All-cellulose Composites, Biodegradation, Mass loss, Microstructures, Soil burial testing.

1. INTRODUCTION

Cellulose is a widely available natural polymer on Earth, consisting of β-1,4-linked glucopyranose units that form a high-molecular-weight linear polymer [1]. The repeating unit of this polymer is a glucose dimer called cellobiose. Cellulose is highly crystalline, fibrous, and insoluble, with exceptional mechanical properties. It is found in wood, cotton, hemp, and cell walls of various plants [2]. It is commonly used in cellulose-based materials due to its availability, low cost, and biodegradability [3]. Its notable properties of cellulose include a density of 1.53–1.89 g/cm^3 [4], tensile strength of 17.8 GPa [5], and Young's modulus of 138 GPa [6]. Cellulose has diverse applications in food, industrial, pharmaceutical, paper, and textile production, as well as in wastewater treatment [7].

Nowadays, a novel class of cellulose-based composites known as all-cellulose composites (ACC) has been developed. It is composed of cellulose for both reinforcing and matrix phases. The use

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of cellulose for both the reinforcing and matrix phases is expected to expedite the biodegradation of ACC [8]. Embedding AKD mitigates the hydrophilicity of cellulose. AKD is a hydrophobic coating due to its long alkyl chains, providing water repellency to cellulose structures [9]. Various studies on AKD-treated cellulose materials have been reported, resulting in excellent water repellency for the materials [10][11][12]. A water contact angle exceeding 100 ° was observed in ocellulose-based materials following AKD treatment [7]. Missoum et al. reported a water contact angle of 100±3˚ in AKD-treated nanofibrillated cellulose (NFC), which correlated with a decrease in water absorption [13]. A similar trend of increased water contact angle and reduced water absorption was also observed in AKD-treated alkali/urea-generated cellulose (AUC) [14]. However, no studies have been published on the biodegradation behavior of AKD-treated ACC.

The development of AKD-treated ACC supports several SDGs by promoting the use of renewable, biodegradable, and high-performance materials. The enhanced properties of these composites, particularly their improved hydrophobicity and durability, make them suitable for water treatment and sustainable material applications, contributing to SDG 6 (Clean Water and Sanitation). Their utilization as bio-based alternatives to petroleum-derived materials supports SDG 9 (Industry, Innovation and Infrastructure) and SDG 12 (Responsible Consumption and Production) by fostering sustainable industrial innovation and resource-efficient production. Furthermore, reducing plastic dependency and environmental pollution aligns with SDG 13 (Climate Action), SDG 14 (Life Below Water), and SDG 15 (Life on Land). Overall, AKD-treated ACC offers a sustainable pathway toward environmentally responsible material development and application.

This study investigates the role of AKD in ACC biodegradation using soil burial testing. It will also control the temperature to examine its effects on the biodegradation of AKD-treated ACC. Several characterization methods were employed, including mass loss measurements, visual assessments, and microstructural analyses. Additionally, a comparative study of the biodegradation rates of cellulose-based materials and the possible mechanisms of biodegradation was discussed.

2. MATERIAL AND METHODS

2.1 Materials

A rayon textile in the form of K2/2 twill weave was used as a precursor material to produce ACC. The textile was based on a multifilament yarn (Cordenka® 700, 2440 dtex, no of filaments: 1350, and final areal weight: 450 g/m^2). Sodium hydroxide (NaOH) (EMSURE®) and urea (HmbG® chemicals) were used as solvents.

2.2 Production of alkyl ketene dimer-treated all-cellulose composite

ACC was produced from a rayon textile *via* solvent infusion processing (SIP), as described by Dormanns et al. [15]. An aqueous solution of NaOH/urea/distilled water (7/12/81 wt.%) was prepared at room temperature using a magnetic stirrer. The rayon textile was cut into a dimension of 160 × 160 mm. The textile was layered on a flat plate and sealed with a vacuum bag. This mould setup was connected to a vacuum pump for the infusion process with a controlled pressure of 19 kPa. The aqueous solution was infused at -12˚C into the rayon textile, wetting it during the infusion process. The setup was disconnected from the vacuum pump and was immersed in 500 ml cooling bath at 0 ± 2˚C for 5 minutes. The partially dissolved cellulose was regenerated and washed using distilled water until the solvent was completely removed. Then, the regenerated cellulose was oven-dried at 60 °C for 24 hours. The sample, identified as ACC, was stored in a sealed plastic bag before further characterisation.

A homogeneous approach was used to produce AKD-treated ACC (Figure 1). AKD coating solution (5 wt.%) was prepared by dissolving AKD in a mixture of ethanol and distilled water at a 4:1 ratio, stirring at 100˚C on a hot plate with a magnetic stirrer until a homogeneous coating solution formed. The regenerated cellulose was dip-coated in 5 wt.% AKD for 1 hour prior to the drying process. Subsequently, the dip-coated regenerated cellulose was dried at room temperature for a while, then further dried in an oven at 100˚C for 1 hour. Finally, the samples were removed from the oven and washed with distilled water to remove any residual AKD from the surface of the formed coated cellulose [16]. The sample, known as AKD-treated ACC, was stored in a sealed plastic bag prior to characterisation.

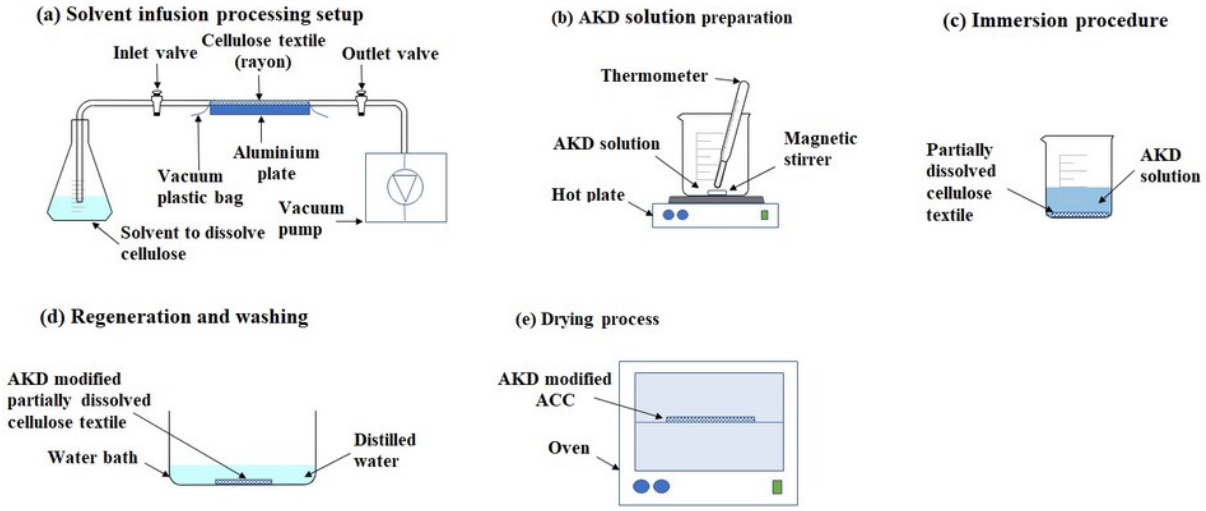

Figure 1: AKD-treated ACC processing route *via* homogeneous approach [7].

2.3 Soil burial testing procedures

Soil burial testing was conducted using a controlled cabinet (Model: FDD-1000, Protech Company, China). ACC and AKD-treated ACC samples were cut into a coupon size of 20 × 80 mm. The samples were preconditioned in an oven at 80˚C for 2 days. The dry mass of the coupons was recorded. The coupons were buried in plant pots, containing compost soil, at a depth of 30 mm. The temperature in the drying cabinet was controlled at two levels of 27±1 and 40±1˚C. The experiment used four plant pots, with three replicates per pot. The soil burial testing was conducted for 7 weeks.

2.4 Material Characterisations

The soil burial testing was conducted to accordance with the previous procedures described by Kalka et al. [17]. The coupons were removed from the soil after 7, 21, 35, and 49 days. The samples were cleaned with distilled water and weighed in the wet state using a lab scale (Model: BSA224S-CW, Sartorius Group, Germany, precision: 0.1 mg) to measure the mass change. The soil will be sprayed with 300 ml of water every day to maintain a constant moisture content. The change of mass is calculated using the following equation (Eq.1).

$$\text{Change in mass (\%)} = \frac{m_d - m_i}{m_i} \times 100 \quad (1)$$

where m_i is the initial mass of dry samples, and m_d is the mass of the samples after a specified time interval. The mass loss indicates the biodegradation rate of the sample. The measurements after the soil burial experiments were based on the dry weight of the samples following collection.

The morphology of the ACC samples was tested after biodegradation in soil by using a scanning electron microscope (SEM) (TM3000, Hitachi, Japan). Then, the sample surface was rapidly coated with platinum film for 120 seconds at 3 mA. The micrographs were acquired using an operating accelerated voltage of 5kV and different magnification options.

3. RESULTS AND DISCUSSION

3.1 The effect of alkyl ketene dimer and temperature on the change in mass

Biodegradation of polymers involves three phases: deterioration, fragmentation, and assimilation/mineralization [18][19]. In the present study, ACC showed significant discolouration (e.g., black spots) at the edges and on the top surface within 49 days of soil burial (Figure 2). At the temperature of 40˚C, the presence of the black spot could be observed as early as day 21, as compared to day 35 for ACC conditioned at room temperature. It was observed that the black spots intensified over time, possibly due to the adsorption of soil particulates or microbial activity, resulting in surface discoloration in the presence of soil moisture. The biodegradation process occurred at a preferred higher temperature, leading to a considerable mass loss. This result suggested that the microorganisms decomposed cellulose at a preferred higher temperature. It was supported by Tremier et al., who reported that microorganisms actively decomposed cellulose at an elevated temperature of 40˚C [20]. This indicated that temperature has huge effects on chemical and biochemical reactions and strongly influences the taxonomic composition and metabolic activities of microbial communities [21]. Another previous study found that a wide range of temperatures between 15-40˚C was used to observe the mass loss of biodegradable plastic *via* soil burial testing, resulting in a greater mass loss at 40˚C [22]. Therefore, the presence of black spots was direct evidence of microbial-induced biodegradation of cellulose. However, no prominent black spots were observed for AKD-treated ACC (Figure 3). This finding might be due to the role of AKD, resulting in phase transformation of cellulose structures, which influences the biodegradation of ACC after the addition of AKD.

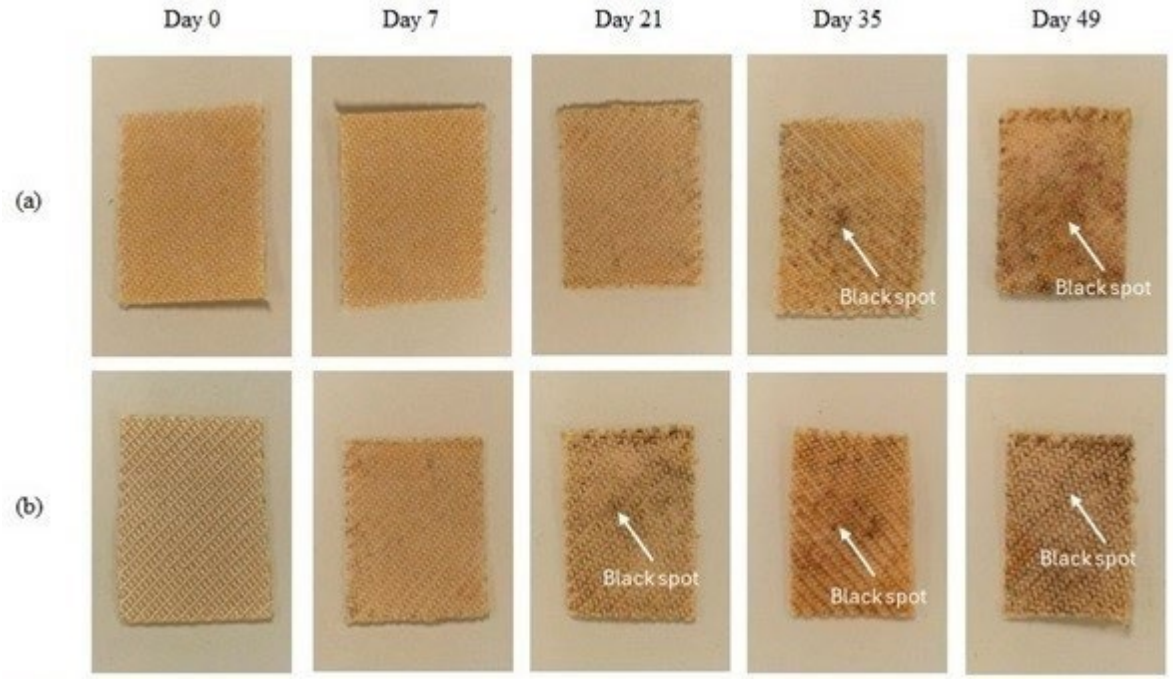

Figure 2: Photograph of ACC before and after soil burial at specified interval time: (a) room temperature, and (b) 40˚C.

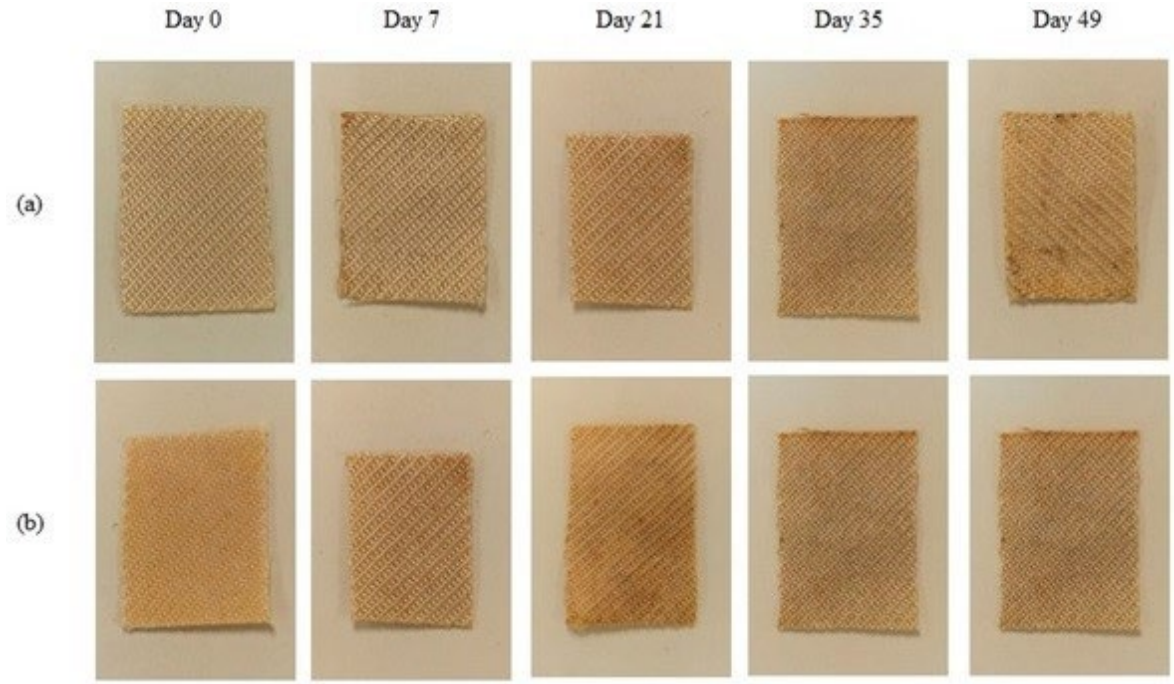

Figure 3: Photograph of AKD-treated ACC before and after soil burial at specified interval time: (a) room temperature, and (b) 40˚C.

The positive change of mass for ACC at room temperature could be observed from 7 to 21 days of soil burial (Figure 4). In contrast, the positive change in mass for ACC at a higher temperature of 40˚C was observed only after 7 days. The positive change of mass was anticipated due to the water absorption during the initial stage of biodegradation. Jiang et al. reported that a higher water absorption promoted surface hydrolysis of the cellulosic fibers, leading to structural plasticisation and performance degradation under the action of water and heat in the soil [23]. These results were supported by previous studies [24][25], highlighting that water favours microbial attack *via* hydrolysis, leading to an increase in the biodegradation rate [26]. It was also reported that water weakened the cellulose materials, increasing their free volume between cellulose chains [27][28]. Water served as a medium for microorganisms to enter the polymer networks, facilitating microbial colonization resulting in inferior mechanical properties [27][29]. It was envisaged that the hydroxyl (OH) group on cellulose structures initiates hydrolysis by absorbing water from the soil, thereby increasing cellulose biodegradation, as indicated by a significant mass loss. It was suggested that increased water absorption during the initial stage of soil burial promoted ACC biodegradation.

In the present study, a shorter time to degradation was observed at higher temperatures. ACC conditioned at 40˚C exhibited a shorter time to degrade at 21 days after soil burial, as compared to ACC conditioned at room temperature, showing time to degrade at 35 days after soil burial (Figure 4). Both ACC conditioned at room temperature and at 40˚C experienced mass loss, as indicated by negative percentage values of 4.6% and 12.2% after 49 days of soil burial, respectively (Figure 4). These results exhibited that biodegradation prevailed in the cellulose structures of ACC. The decomposition of cellulose at the molecular level occurred *via* proteolysis or hydrolysis, involving enzyme activity or fission by water molecules, respectively [30]. This finding was due to enzymes actively breaking down the complex cellulose chains into simpler sugars that microbes can utilize for energy and growth [31]. In addition, thermophilic microorganisms are specifically adapted to thrive at warmer temperatures and become more active at 40˚C [31]. These microbes may even have specialized enzymes specifically tailored for efficient cellulose degradation at higher temperatures. In general, the increase in biodegradation at elevated temperatures suggested that the microorganisms were more active at higher temperatures [32]. The highest degradation rate of cellulose corresponded with a maximum in

the growth rates of soil-inhabiting microbial communities known to occur in the range of 25–30˚C [32].

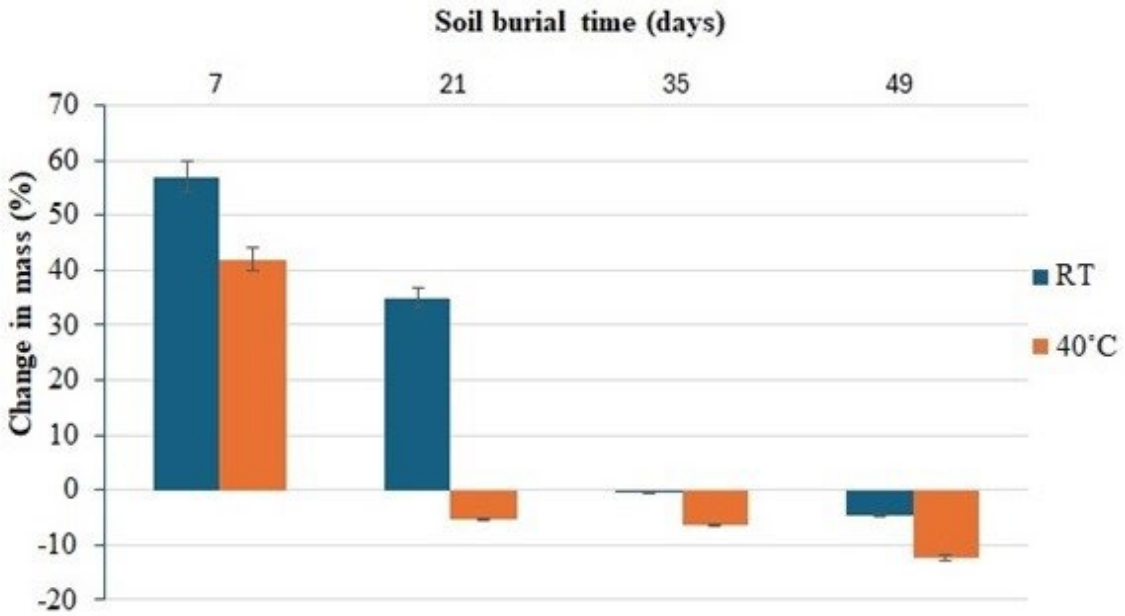

Figure 4: Change in mass of ACC at room temperature and 40˚C as a function of soil burial time. The error bars indicate 5%.

Figure 5 illustrates the positive change in mass of AKD-treated ACC at room temperature and 40˚C as a function of soil burial time. The positive change in mass was due to the water absorption during the initial stage of biodegradation. This trend was observed to range from 40-27% to 31-2.7% at room temperature and 40˚C, respectively. A rapid mass decrease was observed in AKD-treated ACC between 7 and 49 days, with a 91.2% mass loss at 40˚C, compared to only 32.5% at room temperature. Higher water absorption might be due to an inhomogeneous AKD coating, resulting from insufficient drying temperature or microstructural damage (e.g., voids and cracks) caused by sample preparation.

Interestingly, no significant mass loss was observed in AKD-treated ACC at both temperatures over 49 days of soil burial. The hydrolysis reaction slowed down, although water absorption was evident. This was probably due to less accessible OH groups on the cellulose structures to interact with water molecules due to embedded AKD. This result was supported by Nguyen et al., who reported that the intensity of the band at 3272 cm^{-1} (corresponding to the stretching vibrations of OH groups) in polyvinyl alcohol/cellulose nanocrystal (PVA/CNC) nanocomposites decreased with increasing AKD content [33]. This indicated that the AKD diffused and covered the PVA chains and CNC [34]. It was envisaged that AKD forms β-ketoester linkages between AKD and the OH groups of cellulose structures in ACC, thereby hampering biodegradation by microorganisms. This result contributed to a slower biodegradation of AKD-treated ACC.

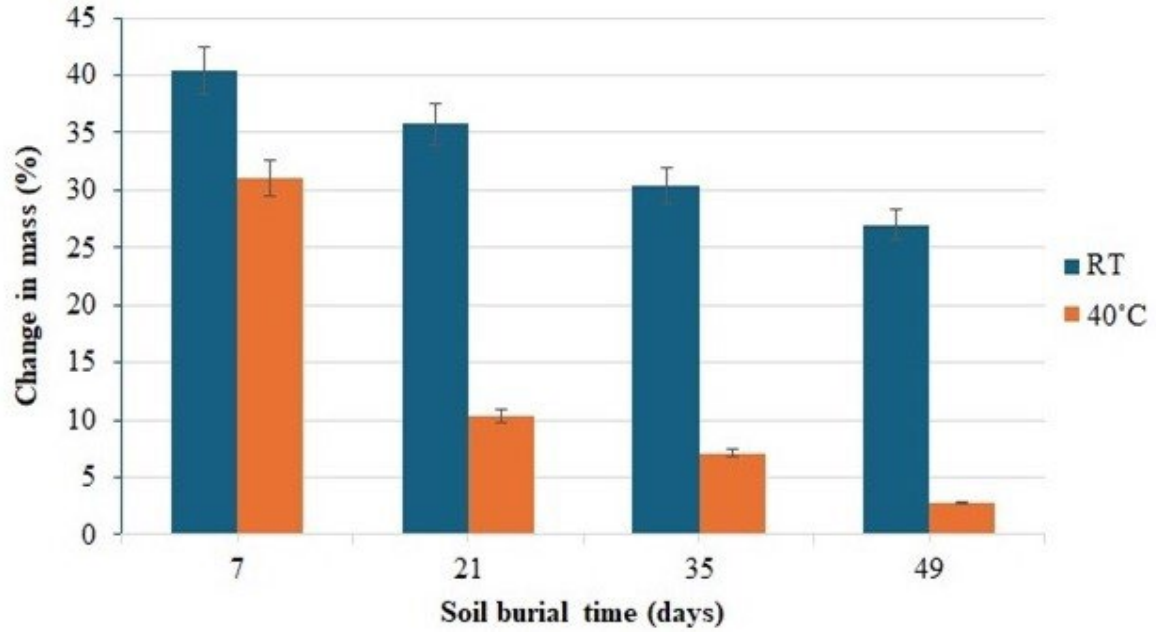

Figure 5: Change in mass of AKD-treated ACC at room temperature and 40˚C as a function of soil burial time. The error bars indicate 5%.

3.2 Microstructural analyses

Figure 6 (a & d) showed that the cellulosic fiber for ACC and AKD-treated ACC was in a uniform structure before soil burial, respectively. It was observed that fiber loosening was prominent for ACC at 40˚C, as indicated by a larger gap and the existence of fiber separation (Figure 6c). This finding correlated with a greater mass loss of 12.2% (Figure 4) and a higher degree of black spots for ACC after 49 days of soil burial at 40˚C (Figure 2b). In contrast, a considerable medium gap was identified for ACC after 49 days of soil burial at room temperature (Figure 6b), resulting in a lower mass loss of up to only 4.6% (Figure 4).

Although small and medium gaps were observed for AKD-treated ACC at room temperature (Figure 6e) and 40˚C (Figure 6f), respectively, negligible mass loss was reported after 49 days of soil burial (Figure 5). This result might be related to no surface discoloration (*e.g.,* black spots) on the AKD-treated ACC surface. It is expected that a longer time to degrade is required for the biodegradation of AKD-treated ACC. This result is probably due to the hydrolysis process, which may take longer for AKD-treated ACC. In addition, the hydrophobic group of AKD is expected to slow biodegradation. This might be due to the formation of β-ketoester bonds between AKD and cellulose structures, as evidenced by the presence of a new peak at 1710 cm^{-1} in Fourier transform infrared (FTIR) spectra [33]. It was also reported that β-ketoester bonds could be formed via covalent or hydrogen bonding with OH groups in cellulose-based materials [7].

It is envisaged that faster biodegradation of ACC was observed compared to AKD-treated ACC (Figure 6e & f), due to the absence of AKD on the cellulose structure, thereby significantly exposing ACC cellulose to microbial activity. The biodegradation of cellulose may be caused by microorganisms (*e.g.*, bacteria and fungi) that produce enzymes capable of breaking down the crystalline structure of cellulose [31]. This finding might be correlated with the formation of filamentous bodies, indicating that cellulose biodegradation actively occurred through microbial activity [35]. Although no filamentous bodies were found for ACC, biodegradation occurred, as indicated by black spots and mass loss. Shiny particles appeared, as shown in Figure 6c & f, probably due to sand particles that adhered to the samples during soil burial testing.

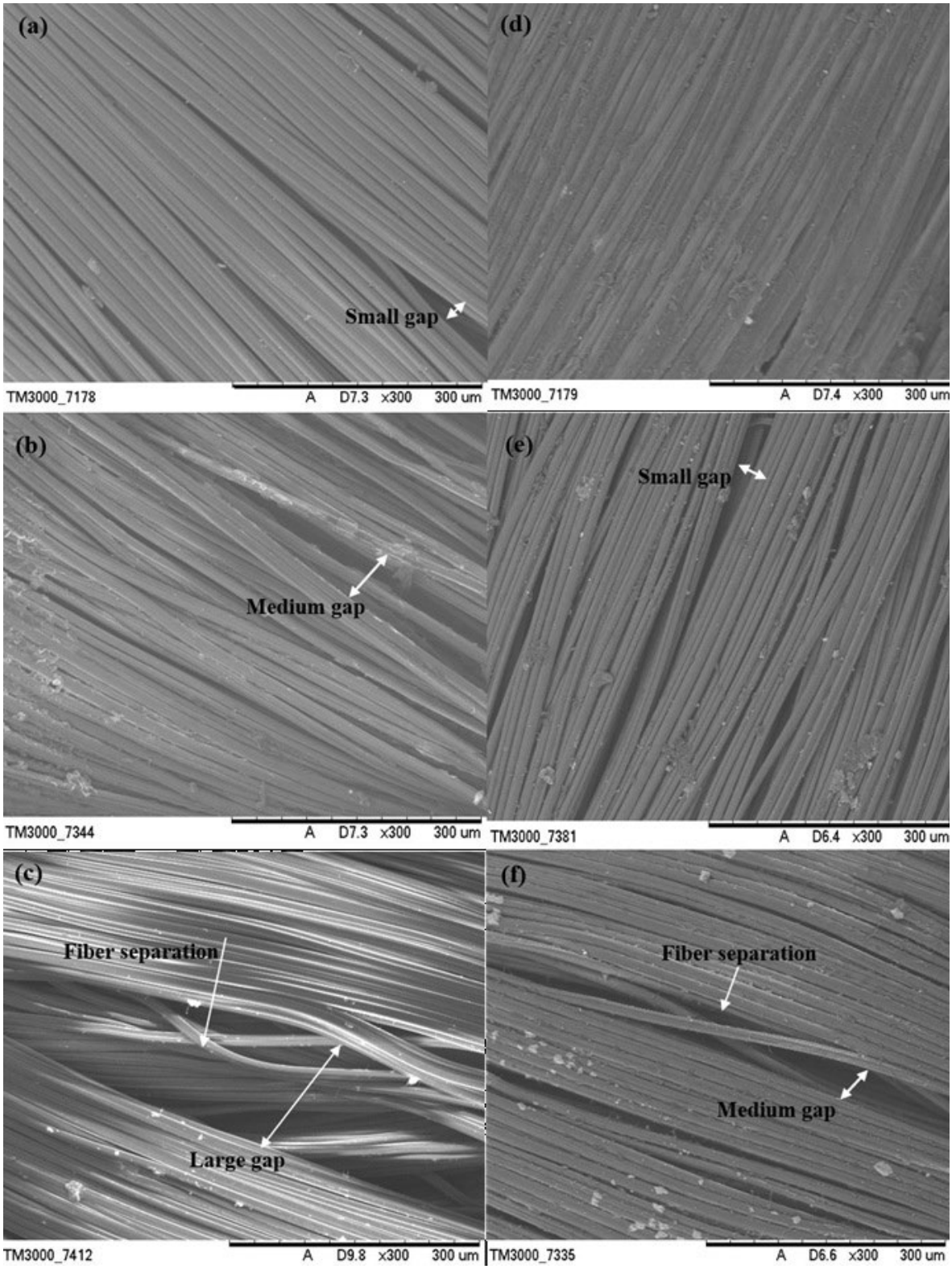

Figure 6: Micrograph images of ACC (a-c) and AKD-treated ACC (d-f), before soil burial (a & d), after 49 days of soil burial at room temperature (b & e), and 40˚C (c & f).

3.3 Comparative studies and biodegradation mechanism of alkyl ketene dimer-treated all-cellulose composite

Table 1 shows an overview of mass loss and time to degrade for various cellulose-based materials (ACC, film, and composite). It could be summarized that the range of mass loss and time to degrade was observed between 2.7 to 100%, and 11 to 180 days, respectively.

Table 1: Overview of mass loss and time to degrade for various cellulose-based materials.

Materials	Types	Mass loss (%)	Time to degrade (days)	Refs.
ACC	ACC	55	70	[35]
Bamboo fiber/Starch	Composite	41	56	[23]
ACC hydrogel	Composite	74	24	[36]
Cotton/starch biocomposite sheet	Composite	80	120	[37]
Starch/NFLC	Film	87	40	[38]
ACC	ACC	18.8	180	[39]
All-cellulose-based film	Film	100	81	[40]
RCF (Durian rind)	Film	82	14	[41]
Viscose (regenerated cellulose)	Composite	9.4	11	[42]
Cellulose film (Banana fiber)	Film	93	28	[43]
PLA/cellulose film	Film	12.3	15	[44]
PVA/CNC/AKD	Composite	17	42	[33]
PLA/CNF/lignin	Composite	2.7	30	[45]
ACC (room temperature)	ACC	4.6	49	Present study
ACC (40˚C)	ACC	12.2	49	Present study
AKD-treated ACC (room temperature)	ACC	-	-	Present study
AKD-treated ACC (40˚C)	ACC	-	-	Present study

Figure 7 illustrates the mass loss and time to degrade for various cellulose-based materials. It was observed that the cellulose film exhibited a greater range of mass loss and a shorter time to degrade compared to other cellulose-based composites (e.g., ACC and the composite). The mass loss and time to degrade for the cellulose film were in the range between 12.3 to 100%, and 14 to 81 days, respectively. Meanwhile, the composite was positioned between the cellulose film and the ACC groups, showing a mass loss range to 2.7 to 80% and a degradation time of 11 to 120 days, respectively. However, the biodegradation of ACC was relatively inferior, as compared to the others. It was shown by lower mass loss and higher time to degrade, 4.6 to 55% and 49 to 180 days, respectively. This might be due to the higher crystallinity of ACC and the source of cellulose used as a precursor material (*e.g.,* rayon).

In this present study, the mass loss for ACC seemed slightly lower (Table 1), as compared to the previous study by Kalka et al. [17]. This result might be due to lower cellulose solubility for ACC produced via NaOH/urea, which contributed to a lower matrix phase generated. Jan et al. reported that the solubility of cellulose in 1-butyl-3-methylimidazolium acetate (BmimAc) and NaOH/urea was >20 wt.%nd 6.5 wt.%, respectively [15]. It was also reported that the matrix phases of ACC produced via BmimAc and NaOH/urea were 10% and 4.6%, respectively, after 60 minutes of dissolution [5][46]. This finding showed that a higher matrix phase in ACC produced *via* BmimAC is evidenced, as compared to ACC produced *via* NaOH/urea. This promoted slower biodegradation for ACC produced *via* NaOH/urea due to the minimal matrix phase generated. It was reported that preferential biodegradation of the matrix phase is expected due to the phase in ACC generally forms with a less ordered structure, as compared with that of the precursor

cellulose fibre, making the material more accessible to microorganisms [35][47]. The matrix phase was amorphous due to preferential dissolution of the crystalline-amorphous series in the cellulose structures, leading to the reassembly of dissolved amorphous onto the lateral surface of neighbouring crystallites, perpendicular to the fiber axis [15].

It was reported that as-received rayon has crystallinity between 32.6 to 42%. [15][48]. Mat Salleh et al. found that the crystallinity of ACC produced via BmimAc increased by up to 58.6% after undergoing dissolution, regeneration, and drying [46]. However, a decrease of crystallinity from 42 to 38.1% was reported for ACC produced via NaOH/urea [15]. This finding indicated that different solvents (*e.g.,* (i) BmimAc, and (ii) NaOH/urea) contributed to a wide range of crystallinity. Although a lower crystallinity was observed for ACC produced via NaOH/urea rather than that of the crystallinity for ACC produced *via* BmimAc, the biodegradation of ACC produced via NaOH/urea was retarded. This result might be due to the less matrix phase and the presence of the β-ketoester bonds, leading to a reduction of microbial activity. This finding was inconsistent with the previous study by Nguyen et al. [33], which reported that the biodegradation rate increased with decreasing crystallinity.

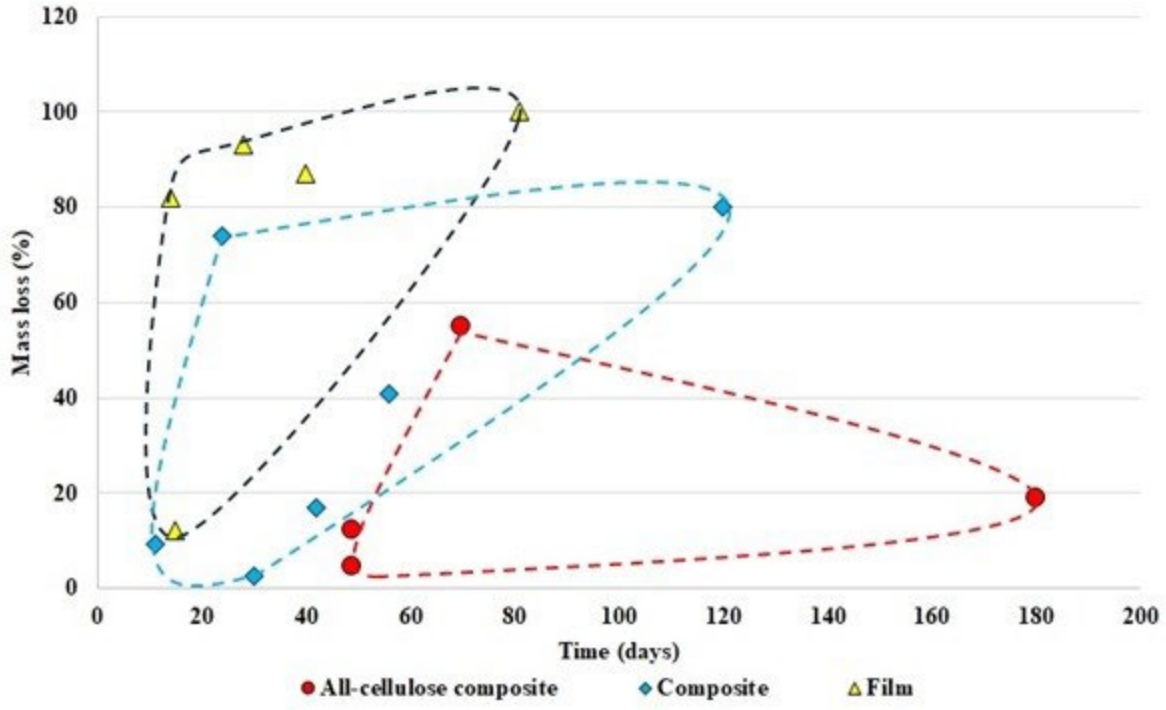

Figure 7: Mass loss and time to degrade for various cellulose-based materials.

The proposed biodegradation mechanism for cellulose-based materials involves biodeterioration, fragmentation, and assimilation, as illustrated in Figure 8. However, in the present study, ACC biodegradation was anticipated to occur only up to Phase 1 (biodeterioration), resulting only in surface discoloration (e.g., black spots) and fiber loosening. This finding was due to the absence of significant fragmentation and assimilation that could be observed within 49 days of soil burial. Water absorption was measured during soil burial, indicating hydrolysis. Torgbo and Sukyai reported that water absorption in cellulose materials caused plasticisation and delamination, leading to a reduction in the glass transition temperature [49]. Rizal et al. found that hydrolysis broke the ester bond prior to microbial activity, thereby lowering molecular weight [18]. It was anticipated that microorganisms attached to the ACC surfaces would exhibit early-stage biodegradation within the ACC, with minimal microbial activity. However, further study is required to examine the glass transition temperature and molecular weight and to provide evidence of hydrolysis during cellulose biodegradation.

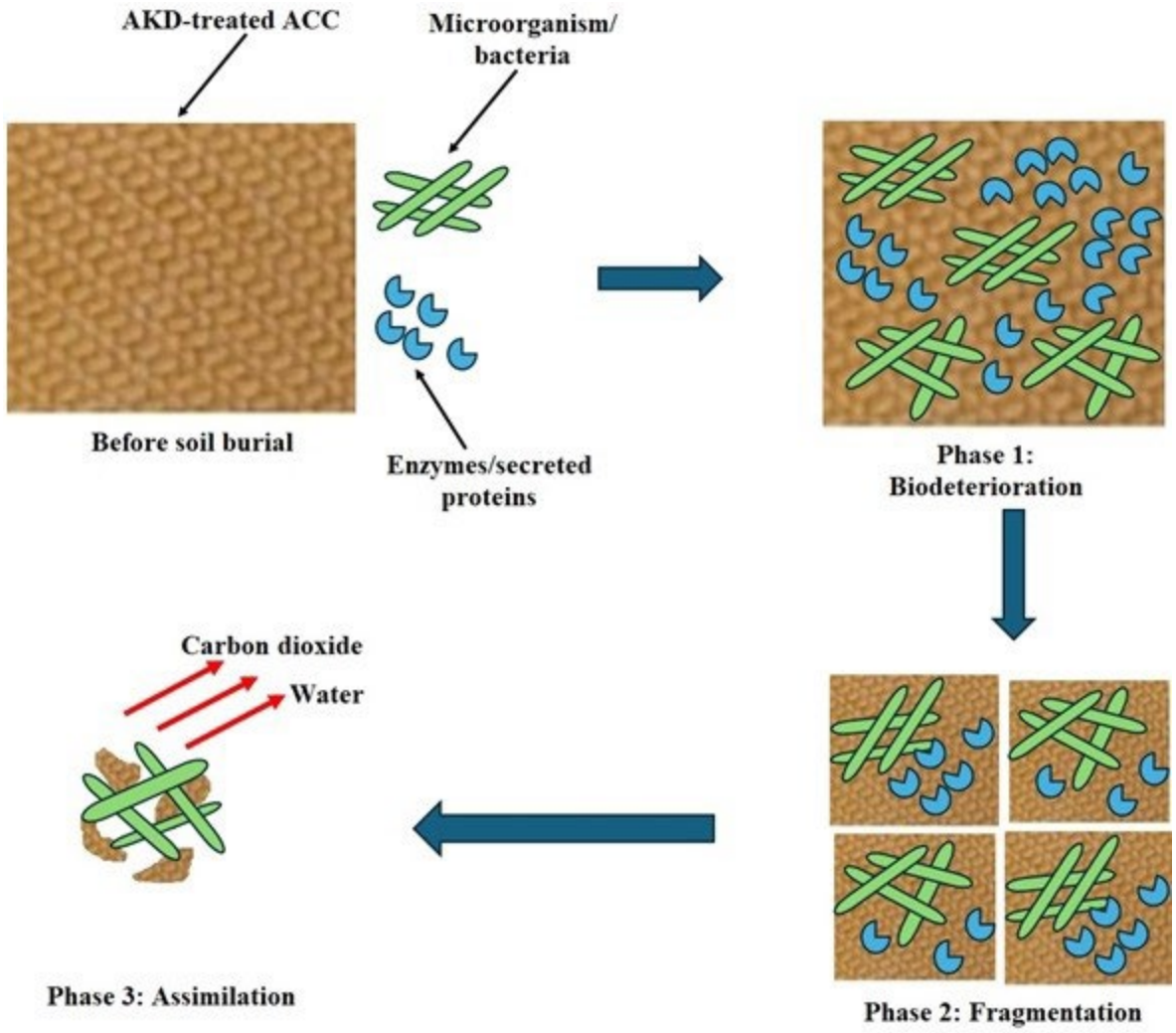

Figure 8: Biodegradation mechanism for cellulose-based materials (*e.g.,* ACC/AKD-treated ACC). Adapted from [18].

4. CONCLUSION

In conclusion, negligible mass loss is investigated for AKD-treated ACC at both temperatures. This is evidenced by the inexistence of black spots and fiber loosening. The addition of AKD seems to retard the biodegradation. This finding may be due to the attachment of AKD's hydrophobic group to cellulose structures. Interestingly, the biodegradation of ACC is observed by a decreasing mass loss up to 4.6 and 12.2% at room temperature and 40˚C, respectively, after 49 days of soil burial. This result was revealed by the presence of fiber loosening and black spots in microstructure analyses. The biodegradation of ACC is likely to be influenced by the solubility of cellulose. Lower cellulose solubility reduces matrix solubility, leading to reduced biodegradation due to the insignificant microbial activity in the matrix phase. Further studies are required to assess the potential for mass loss in AKD-treated ACC with increasing soil burial time. Other characterisations, including X-ray diffraction (XRD), FTIR, and tensile testing, should be carried out to gain deeper insights into the biodegradation of ACC or AKD-treated ACC in the future.

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