

Investigation of the physicochemical, thermal, and optical properties of polyvinyl alcohol/gambir extract biocomposite films

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ABSTRACT: Biodegradable polymer films with improved surface, thermal, and UV-protective properties are increasingly needed for sustainable packaging and protective material applications. In this study, polyvinyl alcohol (PVA) and gambir extract were combined to develop biocomposite films through drying without heating and with different heating durations of 10, 20, and 40 min. The heating duration effect on the morphology, elemental composition, functional groups, crystallinity, wettability, porosity, thermal behavior, and optical properties of the films were systematically investigated. Surface morphology analysis revealed that the sample with 40 min heating duration exhibited a smoother and more homogeneous surface than the unheated film. Elemental analysis confirmed that all films were mainly composed of carbon and oxygen, while Fourier transform infrared analysis showed similar functional group patterns for all samples. The crystallinity index increased with increasing heating duration, indicating enhanced structural ordering. Water contact angle measurements showed that longer heating durations reduced the hydrophilic nature of the films, with the 40 min-heated sample exhibiting the highest contact angle. In addition, this sample showed a smaller average pore diameter, lower glass transition temperature, higher enthalpy of fusion at melting temperature, and higher thermal decomposition temperature. Optical analysis further demonstrated that the PVA/gambir films provided good UV protection, particularly in the UV-B region. Overall, increasing the heating duration, especially to 40 min, improved the physicochemical, thermal, and optical performance of the PVA/gambir biocomposite films.

KEYWORDS: biocomposite, PVA, gambir extract, heating time

INTRODUCTION

Plastic products play an essential role in modern life and industrial development [1]. According to the Organisation for Economic Co-operation and Development (OECD), global plastic production reached 459.75 million tons in 2019 and is projected to increase threefold by 2060. The packaging (40%), building (20%), and automotive (8%) sectors are the major consumers of plastics, along with the household appliance industry [2]. Petroleum-based plastics, such as polyethylene, polyvinyl chloride, and polypropylene, have been widely used in a broad range of applications because of their excellent performance [3]. However, the persistence of these non-biodegradable materials has become a major source of environmental pollution [4]. In this context, biodegradable polymeric materials have emerged as promising alternatives because of their environmental compatibility, abundant availability, and sustainability [4]. Among biodegradable synthetic polymers, polylactic acid (PLA), polycaprolactone (PCL), and polyvinyl alcohol (PVA) have received

considerable attention [5]. In particular, PVA has been widely explored as a substitute for petroleum-based, non-degradable materials [6].

PVA is a water-soluble, non-toxic, and biodegradable polymer with high transparency. It also possesses a large specific surface area, readily form crosslinks with other materials [7], and exhibits excellent film-forming ability [8]. Owing to these advantages, PVA-based materials have been widely applied in food packaging, construction, electronics, printing, coatings, textiles, cosmetics, and paper industries [9]. Despite these merits, the practical use of PVA is still constrained by its relatively poor mechanical performance [1], limited thermal resistance, insufficient ultraviolet (UV) shielding, and relatively high cost [10]. To address these limitations, various fillers have been incorporated into PVA matrices, including starch, chitosan, glycerol, methylcellulose, nanocellulose, wood waste, protein hydrolysates, and plant extracts, to improve its functional properties [11, 12]. For example, the incorporation of *Caesalpinia ferrea* Martius extract into PVA has been reported to enhance the mechanical,

thermal, and antioxidant properties of the resulting films [13]. Plant-derived extracts are particularly attractive because they contain phenolic compounds with strong antioxidant activity [14]. In addition, phenolic compounds from natural extracts can act as effective UV-barrier agents in active films, making them suitable for packaging light-sensitive products such as fatty foods [15].

Gambir extract is an environmentally friendly polyphenolic material obtained from the leaves of the gambir plant [16]. Gambir grows naturally in several regions of Indonesia, particularly in Sumatra, Java, and Bali [17]. Historically, gambir has been an important trade commodity in Indonesia since the early nineteenth century [16]. Pure gambir is composed predominantly of catechins, which are phenolic compounds [18]. It also contains a small amount of quercetin, a pigment responsible for the characteristic yellow color [19]. Previous studies have shown that incorporating gambir extract into PVA films improves mechanical properties, thermal stability, antibacterial activity, and UV protection compared with neat PVA [6]. The high catechin content of gambir extract contributes to its ability to absorb UV radiation, thereby enhancing the UV-protective performance of PVA/gambir films [20]. In addition to filler incorporation, processing conditions during film preparation can also influence the characteristics of PVA films and ultimately determine their final properties [21].

Processing conditions are known to play a crucial role in determining the structure and properties of a material [22]. Among these conditions, drying is one of the most important steps in film preparation. Previous studies have shown that differences in drying methods and conditions significantly affect the physical properties of chitosan films and the release behavior of phenolic compounds incorporated into the matrix [23]. Drying conditions can be controlled through parameters such as heating temperature and duration. In film fabrication, drying time has been reported to strongly influence the physical and chemical characteristics of the final film [24]. Moreover, optimizing of the drying process is important for improving operational efficiency in terms of both processing time and cost. However, the effect of heating duration during the drying process of PVA/gambir films has not been widely investigated. Therefore, this study aims to characterize PVA-based films containing gambir extract prepared under different drying conditions, namely drying without heating and drying with heating durations of 10, 20, and 40 min.

MATERIALS AND METHODS

Preparation of PVA/gambir film

Dried gambir powder (Sumatran Biota Laboratory, Indonesia) was dispersed in distilled water at a ratio of 1:100 in a 250 ml Pyrex glass beaker. The suspen-

sion was stirred using a magnetic stirrer at 50 °C and 500 rpm for 30 min. It was then centrifuged at 500 rpm for 30 min, after which the supernatant was separated from the residue. PVA with a molecular weight of approximately 75,000 g/mol and a hydrolysis degree of > 99% (Sigma-Aldrich, Germany) was added to the residue-free gambir solution at a ratio of 1:10. The mixture was stirred at 80 °C and 500 rpm for 2 h until gelation occurred. The resulting PVA/gambir gel was subsequently ultrasonicated (SJIA 1200 W, Shuangjia Instrument, China) at 600 W for 5 min and then poured into a Petri dish. The unheated PVA/gambir film samples were dried at room temperature. For the heating treatment, samples were dried in a vacuum oven at 100 °C for 10, 20, or 40 min. The samples prepared for characterization were labeled A (no heating), B (10 min heating), C (20 min heating), and D (40 min heating).

Characterization of PVA/gambir film

Surface morphology of the PVA/gambir film samples was examined using scanning electron microscopy (SEM; Inspect-S50, FEI, USA) at a magnification of 1 k \times . The elemental composition of the PVA/gambir films was analyzed using energy-dispersive X-ray spectroscopy (EDX; AMETEK EDAX TSL, USA). Functional groups in the PVA/gambir films were identified using Fourier transform infrared spectroscopy (FTIR; IRPrestige-21, Shimadzu, Japan) over a wavenumber range of 400–4000 cm⁻¹. The crystallinity index (CI) of the PVA/gambir films was determined using X-ray diffraction (XRD; X'Pert PRO, PANalytical, UK) at a diffraction angle of 10–90° (2θ) and $\lambda = 1.54$. The CI was calculated using Eq. (1) [23],

$$CI = \frac{I_f - I_s}{I_f} \quad (1)$$

where I_f is the intensity of the fundamental band peak at $2\theta = 18.5\text{--}20^\circ$ and I_s is the intensity of the secondary band peak at $2\theta = 28.0\text{--}28.5^\circ$. The water contact angle of the PVA/gambir films was measured according to ASTM D7334 with the aid of ImageJ software. A 10 μ l water droplet was deposited onto the film surface using a micropipette, and the droplet image was captured immediately upon contact with the film surface. The contact angle of each film was measured at three different points, and the average value was reported. The porosity of the PVA/gambir films was analyzed using Brunauer–Emmett–Teller (BET) analysis (TriStar II Plus 3020, Micromeritics, USA). The melting temperature and enthalpy of the samples were determined by differential scanning calorimetry (DSC), while the thermal decomposition behavior of the PVA/gambir films was evaluated by thermogravimetric analysis (TGA), using a TGA/DSC 1 instrument (Mettler Toledo, Switzerland). For both analyses, approximately 20 mg of sample was placed

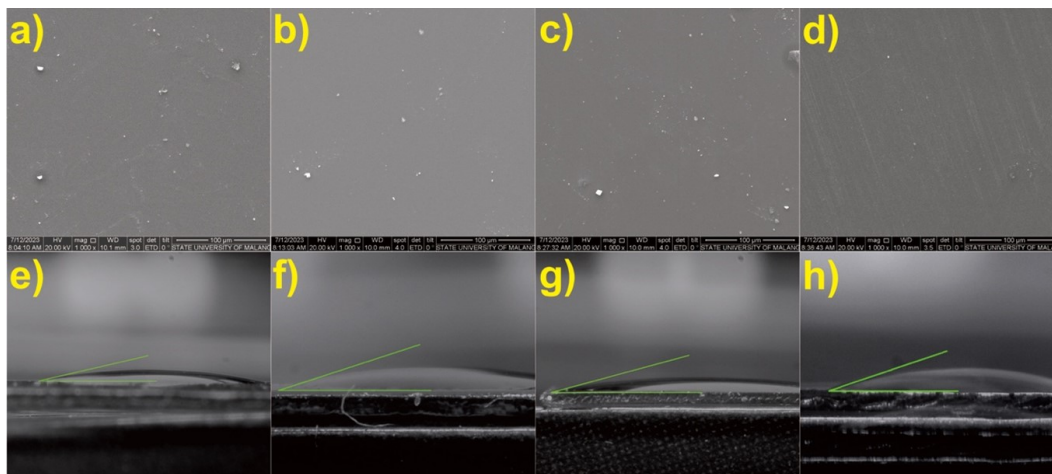


Fig. 1 Surface morphology and water contact angle of PVA/gambir film samples under different heating durations: (a, e) no heating, (b, f) 10 min, (c, g) 20 min, and (d, h) 40 min.

in a crucible. The DSC analysis was performed from 30 to 350 °C at a heating rate of 10 °C/min, whereas the TGA analysis was carried out from 30 to 800 °C at the same heating rate. UV-protection properties were evaluated using a UV-Vis spectrophotometer (Specord 200 Plus, Analytik Jena, Germany) over a wavelength range of 200–1100 nm. The samples were prepared in dimensions of 50 mm × 50 mm.

RESULTS AND DISCUSSION

Surface morphology

SEM analysis was performed to examine the surface morphology of the PVA/gambir film samples. Fig. 1a–d shows the surface morphology of the PVA/gambir films at a magnification of 1 k×. The sample dried without heating exhibited a rough and uneven surface morphology with the presence of round pores. This may be attributed to oxygen trapped in the sample during the film-forming process. In addition, the incorporation of gambir extract may also influence the surface roughness of the PVA film because of the interaction between PVA molecules and gambir extract [18]. The PVA/gambir films samples heated for 10 and 20 min also showed rough and uneven surfaces, although no pores were observed, unlike in the unheated sample. The rough and uneven morphology of these films may be associated with water evaporation during the heating process, which can lead to surface irregularities [24]. In contrast, the PVA/gambir film heated for 40 min exhibited a smoother and more homogeneous surface morphology than the films dried without heating or heated for 10 and 20 min. These results suggest that increasing the heating duration can improve surface smoothness. However, the surface of PVA films remains smooth and homogeneous only up to a certain heating temperature and duration; beyond

these conditions, thermal degradation may occur and hinder the formation of more regular structural units [25].

Elemental contents

EDX analysis was performed to determine the elemental composition of the PVA/gambir film samples, and the results are presented in Table S1. The analysis showed that all samples were mainly composed of carbon and oxygen, with carbon being the dominant element. The presence of carbon and oxygen in all PVA/gambir film samples can be attributed to the chemical structure of PVA [24]. The carbon content slightly decreased from 75.8 wt% in the unheated sample to 75.6 wt%, 75.5 wt%, and 75.1 wt% after heating for 10, 20, and 40 min, respectively. In contrast, the oxygen content showed a slight overall increase, changing from 24.0 wt% in the unheated sample to 24.2 wt%, 24.0 wt%, and 24.7 wt% in the heated samples. These results indicate that the heating duration during the drying process had minimal effect on the overall elemental composition of the PVA/gambir films.

Functional groups

As shown in Fig. 2a, the FTIR spectra of the PVA/gambir film samples several characteristic absorption peaks. The peaks at 3643.53 cm^{-1} and 2954.94 cm^{-1} were attributed to O–H and C–H stretching associated with water uptake [6]. The O–H stretching observed in this region may also be related to strong intramolecular hydrogen-bond interactions [25]. The absorption band at 2400–2300 cm^{-1} was assigned to carbon dioxide present in the atmosphere [26]. Furthermore, the peaks at 1249.87 cm^{-1} and 1118.71 cm^{-1} were associated with C–O stretching vibrations in the 1000–1300 cm^{-1} region, which are related to vibrations in

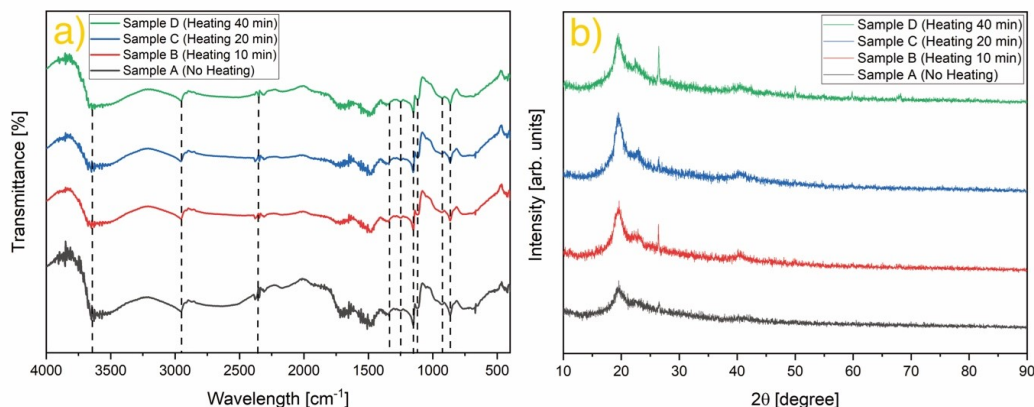


Fig. 2 (a) FTIR and (b) XRD graph of PVA/gambir film samples.

the catechin group [25]. The peak at 1151.50 cm^{-1} was attributed to the formation of crystalline bands in PVA [25]. In addition, the absorption peak at 927.76 cm^{-1} was associated with C–OH and C–O stretching in phenolic compounds [27]. The final peak, located at 864.11 cm^{-1} , was attributed to C–C vibrational modes in the PVA main chain [27].

Crystallinity index

XRD analysis was performed to determine the crystallinity index of the PVA/gambir film samples. Fig. 2b shows the XRD patterns of the PVA/gambir films. As shown in Fig. 2b, the unheated PVA/gambir film (sample A) exhibited three characteristic diffraction peaks. One strong peak was observed at $2\theta = 19.5^\circ$, along with two weaker peaks at $2\theta = 26.53^\circ$ and 41.1° , which were assigned to the (101), (101), and (202) lattice planes, respectively [6]. The prominent peaks at $2\theta = 19.5^\circ$ and 26.53° can be attributed to strong inter- and intramolecular hydrogen bonding between the OH groups of the PVA chains [28]. According to El-Shamy and Zayied [29], the peak at 19.5° corresponds to the crystalline region of the primary semi-crystalline PVA structure, which arises from inter- and intramolecular hydrogen bonding within the PVA molecular chains. Generally, all PVA/gambir film samples showed similar characteristic peaks at approximately $2\theta = 19^\circ$ and 26° , corresponding to the (101) crystal plane of PVA, confirming the semi-crystalline nature of the material.

Based on the data presented in Table S2, the CI increased progressively with heating duration, reaching a maximum of 65.35% for sample D, indicating the formation of more ordered structural domains in the PVA/gambir films. This increase may be attributed to the formation of more ordered structural units in the PVA/gambir films as a result of prolonged heating. A higher degree of crystallinity indicates greater regularity in the molecular arrangement of the sample [28]. In addition, heat treatment at different durations may reduce the water content of the sample, thereby

increasing polymer chain ordering through the formation of polyene structures [25].

Water contact angle

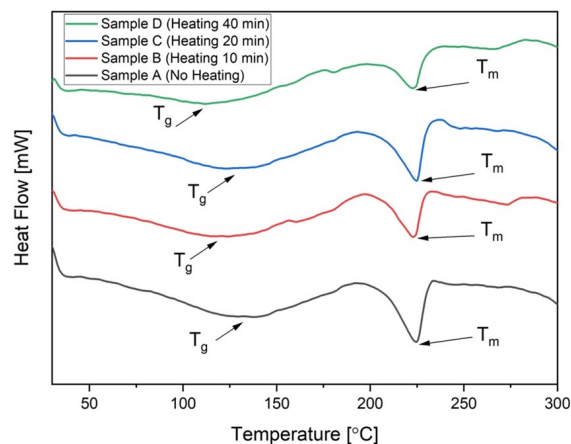
Water contact angle analysis was used to evaluate the hydrophilicity of the PVA/gambir film samples. Fig. 1e–f and Table S3 present the water contact angle results for the PVA/gambir films. As shown in Table S3, all samples were classified as hydrophilic materials. The contact angle of hydrophilic materials ranges from $0^\circ < \theta < 90^\circ$, whereas that of hydrophobic materials ranges from $90^\circ < \theta < 180^\circ$ [30]. A higher water contact angle indicates that the sample is less hydrophilic. In this study, the water contact angle increased with increasing heating duration, with sample D exhibiting the highest average value of 18.74° . This behavior may be attributed to the effect of heating, which reduce the number of free hydroxyl groups and increase the crystallinity of PVA, thereby leading to a higher contact angle [31].

Porosity

BET testing was conducted to identify porosity in the PVA/gambir film samples. Table 1 presents the surface area of the PVA/gambir film samples. Based on Table 1, the sample surface area and total pore volume in the PVA/gambir film show an increase in surface area and pore volume with increasing heating duration. However, a decrease occurred in the average pore diameter of the PVA/gambir film samples with increasing heating duration. The decrease in pore diameters is attributed to the increase in crystalline area which makes the PVA/gambir film samples denser due to heating [32]. Pore size is classified according to the International Union of Pure and Applied Chemistry (IUPAC), namely micropores (diameter (d) $< 2\text{ nm}$), mesopores ($2\text{ nm} < d < 50\text{ nm}$), and macropores ($d > 50\text{ nm}$) [33]. Based on the pore diameter size, all PVA/gambir film samples can be classified as mesoporous adsorbents.

Table 1 Average pore diameter, surface area and total pore volume of PVA/gambir film samples.

Sample name	Average pore diameter [nm]	BET surface area [m ² /g]	Total pore volume [m ³ /g]
Sample A (no heating)	6.9128	1.3804	0.002386
Sample B (heating 10 min)	5.9038	1.4778	0.002181
Sample C (heating 20 min)	5.5603	2.0572	0.003040
Sample D (heating 40 min)	5.3760	2.4078	0.003236

**Fig. 3** DSC graph of PVA/gambir film samples.**Table 2** Glass transition temperature (T_g), melting temperature (T_m), and enthalpy of fusion at melting temperature (ΔH_m) of PVA/gambir film.

Sample name	T_g [°C]	T_m [°C]	ΔH_m [J/g]
Sample A (no heating)	133.16	222.87	45.5
Sample B (heating 10 min)	120.46	221.61	43.37
Sample C (heating 20 min)	119.27	223.12	50.33
Sample D (heating 40 min)	110.55	221.54	47.34

Based on Table 1, the highest surface area and pore volume are in the PVA/gambir film sample with 40 min heating with values of 2.4078 m²/g and 0.003236 cm³/g, respectively. These surface area and total pore volume values are higher than the unheated PVA/gambir film samples with 1.3804 m²/g and 0.002386 cm³/g values, respectively. These results demonstrate that heating duration influences pore formation, thereby increasing both surface area and pore volume. This increase in surface area can offer more surface-active sites, resulting in increased adsorption capacity efficiency [29]. Additionally, the heating also performed on the film/gambir samples has an impact on the water content of the samples so that it can affect the porosity value [34].

Melting temperature and enthalpy

DSC testing was conducted to identify the glass transition temperature (T_g), melting temperature (T_m), and enthalpy of fusion at melting temperature (ΔH_m)

in the PVA/gambir film sample. Fig. 3 and Table 2 show the DSC result of the PVA/Gambir film sample. The graph in Fig. 3 shows T_g , T_m , and ΔH_m of the PVA/gambir film samples. The decrease or shift in T_g value occurs as the heating duration of the sample increases and can be attributed to the evaporation of water molecules in the sample [24]. Heating performed on the PVA/gambir film sample with a long duration can cause faster evaporation of water molecules, thus affecting the T_g value of the PVA/gambir film sample. Not only that, the decrease in T_g value can be attributed to the reduction of hydrogen bonds in the same amorphous domain [35].

Based on Table 2, there is no significant increase in T_m value as the heating duration increases. The decrease in T_m value that occurs in the PVA/gambir film samples in sample C and sample D can be attributed to the degradation of the polymer structure in PVA [24]. The decrease in ΔH_m value can be attributed to the decrease in intermolecular interactions in the compound [36] and can also signify the amorphous nature of the composite [24]. A greater amount of energy is essential to break the strong hydrogen interactions formed between the hydroxyl groups of the PVA chain and melt the crystallites [36].

Decomposition rate identification

TGA analysis was performed to evaluate the thermal decomposition behavior of the PVA/gambir film samples. Fig. 4 and Table S4 show the decomposition profiles of the PVA/gambir films. In Fig. 4a, all PVA/gambir film samples exhibited mass loss in three stages with increasing temperature. The first stage of mass loss occurred in the range of 65–150°C and was attributed to the evaporation of water molecules present in the PVA/gambir films [6]. The second stage of mass loss occurred at temperatures between 300 and 450°C. This stage was associated with the decomposition of PVA and gambir [6]. In addition, the mass reduction in this stage may also be related to the loss of –OH groups and the deacetylation of the PVA chains [37]. At temperatures above 450°C, no significant residual mass was observed, indicating nearly complete combustion of the PVA/gambir films and their final decomposition into ash [38]. The maximum decomposition temperature (T_{max}) of the PVA/gambir film samples during the second stage of mass loss is presented in Table S4. The increase in T_{max} with heating treatment suggests that the drying

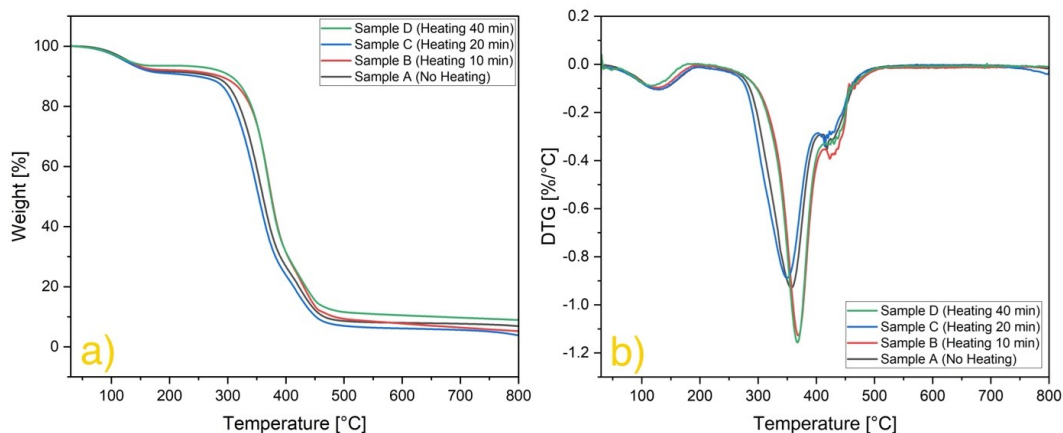


Fig. 4 (a) TGA and (b) DTG graphs of PVA/gambir film samples.

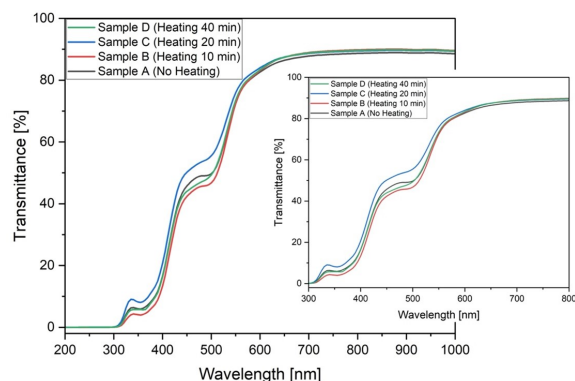


Fig. 5 Light transmission chart of PVA/gambir film samples.

process influenced the decomposition behavior of the PVA/gambir films. The observed trend indicates that a higher T_{\max} contributed to improved thermal stability [38].

UV protection identification

UV-Vis analysis was conducted to evaluate the UV-protection properties of the PVA/gambir film samples. Fig. 5 and Table S5 present the light transmittance of the PVA/gambir films. Based on Fig. 5, the PVA/gambir film heated for 10 min exhibited the highest UV light absorption, transmitting 13.3% of UV light at a wavelength of 400 nm (Table S5). However, UV light transmittance increased with further heating, as observed for sample C (20 min heating) and sample D (40 min heating). This result indicates a decrease in UV absorption capacity in both samples. Solar UV radiation divided into three regions: UV-C (220–280 nm), UV-B (280–320 nm), and UV-A (320–400 nm) [39]. All PVA/gambir film samples exhibited good UV-blocking performance as shown in Fig. 5. All samples can be considered to provide UV-B protection because no light transmission was observed below 280 nm [6]. These

results suggest that increasing the heating duration beyond 10 min does not significantly enhance the UV-protection performance of the films.

Advantages of adding gambir extract to PVA Film

Pure PVA film has been widely used in various applications, but it has certain drawbacks, such as UV protection, thermal properties, and hydrophilicity [40]. The use of bio-based materials has been widely adopted, such as the use of pineapple peel extract, *Caesalpinia ferrea* Martius plant extract, *Lavandula angustifolia* plant extract, *Spatholobus littoralis* Hassk plant extract, and *Calendula officinalis* extract [12, 41–44], and have been proven to enhance the properties of PVA films. The use of gambir extract in this study improved the UV protection properties, it is also an effort to reduce environmental pollution caused by excessive use of synthetic polymers. Additionally, the processing of PVA/gambir films showed changes, particularly in hydrophilicity and thermal properties (as shown in Table 3). The improved properties of PVA/gambir films (heated for 40 min) have potential applications in various fields, particularly food packaging.

CONCLUSION

This study investigated the effect of drying treatment on PVA/gambir films prepared without and with heating for 10, 20, and 40 min. The results showed that increasing the heating duration influenced the surface, wettability, UV-protection, and thermal properties of the films. In particular, prolonged heating reduced the hydrophilic nature of the films, improved their thermal stability, and maintained good UV-blocking performance. The UV-protection ability of the PVA/gambir films can be attributed to the phenolic compounds, particularly catechins, in the gambir extract. Among all samples, the PVA/gambir film heated for 40 min exhibited the best overall performance, showing a water contact angle of 18.74°, providing UV-B protec-

Table 3 Comparison of the properties of PVA/gambir film with other PVA films.

Sample	CI (%)	WCA (°)	T _{max} (°C)	UV-Vis	Ref.
PVA/gambir (heating 40 min)	65.4	18.74	368.4	UV-B	This work
PVA (pure)	43.0	–	304.6	UV-C	[6]
PVA (pure)	27.7	–	312.0	UV-C	[18]
PVA (pure)	–	–	332.3	UV-C	[40]
PVA (pure)	–	20.39	–	UV-C	[41]
PVA/ <i>Caesalpinia ferrea</i> Martius extract (5%)	–	–	346.0	–	[13]
PVA/ <i>Lavandula angustifolia</i> extract (5%)	–	57.67	–	UV-B	[42]
PVA/Corn strach/ pineapple peel extract (5%)	–	–	377.0	–	[43]
PVA/bajakah tampala (<i>Spatholobus littoralis</i> Hassk) extract (2%)	–	41.68	–	UV-A	[41]
PVA/CMC/ <i>Calendula officinalis</i> extract (0.1%)	–	16.80	–	–	[44]

tion, and exhibiting a superior thermal property with a T_{max} value of 368.4°C. These findings suggest that the PVA/gambir film prepared with a heating duration of 40 min has strong potential for various applications, particularly as an environmentally friendly food-packaging film with good UV resistance.

Appendix A. Supplementary data

Supplementary data associated with this article can be found at <https://dx.doi.org/10.2306/scienceasia1513-1874.2026.031>.

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Appendix A. Supplementary data**Table S1** Elemental content of PVA/gambir film.

Sample name	Element	Weight %
Sample A (no heating)	C K (Carbon)	75.8
	O K (Oxygen)	24.0
Sample B (heating 10 min)	C K (Carbon)	75.6
	O K (Oxygen)	24.2
Sample C (heating 20 min)	C K (Carbon)	75.5
	O K (Oxygen)	24.0
Sample D (heating 40 min)	C K (Carbon)	75.1
	O K (Oxygen)	24.7

Table S2 Elemental content of PVA/gambir film.

Sample name	Crystallinity index (CI) [%]
Sample A (no heating)	53.51
Sample B (heating 10 min)	59.72
Sample C (heating 20 min)	63.36
Sample D (heating 40 min)	65.35

Table S3 Water contact angle of PVA/gambir film.

Sample name	Average value of water contact angle [°]
Sample A (no heating)	15.39
Sample B (heating 10 min)	17.89
Sample C (heating 20 min)	18.47
Sample D (heating 40 min)	18.74

Table S4 Temperature of decomposition rate of PVA/gambir film.

Sample name	T _{max} [°C]
Sample A (no heating)	358.1
Sample B (heating 10 min)	369.1
Sample C (heating 20 min)	349.4
Sample D (heating 40 min)	368.4

Table S5 Light transmission of PVA/gambir film.

Sample name	Light transmittance [%] at 400 nm
Sample A (no heating)	16.7
Sample B (heating 10 min)	13.3
Sample C (heating 20 min)	20.8
Sample D (heating 40 min)	17.3