Convertibility and Water Resistance of Wax-based Spray Coating with the Addition of Polylactic Acid

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Petroleum-based plastic coatings are used to create moisture barriers and heat-sealable layers on paperboard. Despite their good convertibility and barrier properties, the use of sustainable biobased polymer coatings as alternatives has attracted interest due to environmental concerns. In this study, the convertibility and water resistance of bio-based multilayered coating composed of biowax and polylactic acid (PLA) were investigated. Convertibility of the coatings was studied through heat-sealing experiments and by evaluating their durability when they were subjected to high stresses during creasing and folding. Surface imaging was performed to evaluate film formation and coating integrity. The wettability and water absorption properties of the coatings were also investigated. Different coating compositions resulted in different film formation processes, surface appearances, and water absorption. The presence of wax in a coating increased its hydrophobicity and reduced its water absorption already with the smallest addition in the coating. However, a high wax content in the coating caused defects in the coating layer, whereas the addition of PLA increased the convertibility of the coated material. This indicates that water resistance, heat sealability, and convertibility can be simultaneously achieved by optimizing the composition of wax and PLA coatings.

DOI: 10.15376/biores.19.4.9631-9644

Keywords: Bio-based coating; Convertibility; Heat sealing; Water resistance

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INTRODUCTION

Fiber-based packaging materials are often coated with petroleum-based plastics to improve their resistance to oxygen, water vapor, and grease and to create heat-sealable layers. However, concerns over the recyclability and biodegradability of plastics have driven the development of more sustainable alternatives for barrier coatings on paper and paperboard. The use of water-based dispersions and biodegradable components in coatings has been investigated for packaging applications. Bio-based coatings can also provide nontoxic alternatives to fluorochemicals, or other harmful substances used to induce barrier properties in coatings or surface modifications (Rastogi and Samyn 2015; Mujtaba *et al.* 2022; Juikar and Warkar 2023).

Plastic coatings provide hydrophobicity and water or moisture resistance to fiber-based materials. Bio-based polymers used as coating components, such as polysaccharides, can provide barrier properties to some extent. However, they are often hydrophilic, and the use of moisture-sensitive materials, such as packaging materials, may be challenging. The moisture sensitivity of such materials can be reduced by hydrophobic or surface roughness

modifications (Zhang et al. 2014; Rastogi and Samyn 2015) or by changing their crystallinity. For example, the properties of polylactic acid (PLA) are affected by the ratio of its isomers; in addition, differences in crystallinity (Rastogi and Samyn 2015) may affect its water vapor permeability (Rastogi and Samyn 2015; Kunam et al. 2022). The use of PLA coatings to improve the water resistance and mechanical properties of fiber-based materials has also been reported (Rhim et al. 2007; Rhim and Kim 2009; Helanto et al. 2022). In addition, inherently hydrophobic materials, such as waxes, can be used as coating components to increase the hydrophobicity and water resistance of coatings (Chiumarelli and Hubinger 2012; Zhang et al. 2014; Wang et al. 2016; Wang et al. 2018). The barrier properties of coatings can be further improved by applying multilayered coating layers (Balan et al. 2015; Koppolu et al. 2019; Lyytikäinen et al. 2021); however, the brittleness of many bio-based coatings during converting remains a challenge (Chiumarelli and Hubinger 2012; Leminen et al. 2015; Rastogi and Samyn 2015; Tanninen et al. 2015).

Cutting, creasing, and folding during the converting process introduce stress in a material, particularly the coating layer. Cutting and creasing are typically performed simultaneously by flatbed die cutting during material processing. Creasing relies on the creation of intentional damage to the paperboard structure. Ideally, the paperboard should undergo internal delamination, which is associated with the breaking of fiber–fiber joints (Marin *et al.* 2022). In addition to stresses generated during creasing, folding poses challenges to the maintenance of coating layer integrity. The coating layer should be able to withstand stress without cracking because defects degrade its barrier properties and appearance. Cracking occurs in the coating layer when excessive stress is applied due to the rigidity of the upper layer in the material (Beex and Peerlings 2009). A previous study on the folding of various creased samples noted that creases do not have to be die cut too forcefully because the desired functionality can be achieved with less force (Tanninen *et al.* 2021). Therefore, the magnitude of the maximum creasing force that can be applied without damaging the coating layer should be determined.

Heat sealability is an important consideration in packaging applications. Conventional fossil-based plastics, such as polyethylene (PE), are often used to create heat-sealable layers on fiber-based materials. Compared with conventional coatings, bio-based coatings often require a higher coat weight or plasticizers to improve heat sealability. Polylactic acid is a bio-based thermoplastic that has been reported to have good mechanical properties, sealability, and seal strength (Auras *et al.* 2004; Rocca-Smith *et al.* 2019; Helanto *et al.* 2021; Ilhan *et al.* 2021; McCurdy *et al.* 2022). However, because heat sealing is generally affected by the temperature, pressure, and time, excessively high temperatures result in PLA film shrinkage and seal deterioration (Auras *et al.* 2004; McCurdy *et al.* 2022). In food packaging applications, a wide sealing temperature range and sealability with short sealing times are desirable properties (Ilhan *et al.* 2021). Even though PLA has good sealing performance, PLA coatings require a moderate coat weight and the use of additives to improve their convertibility because PLA is more brittle and stiffer than the more typically used PE (Helanto *et al.* 2021; McCurdy *et al.* 2022).

Although the barrier and mechanical properties of PLA and wax in coatings on fiber-based materials have been investigated previously, the mechanical durability of PLA-and wax-containing coatings during converting processes should be further clarified. The aim of this study is to investigate the effects of water-based PLA dispersions on the convertibility, heat sealability, and water resistance of wax-based coatings. Cutting, creasing, and folding were chosen as the converting methods for investigation because of the high demands they place on the properties of the coating layers. Spray coating was used

as the method to achieve a multilayer coating with specific coat weights on a paperboard substrate.

EXPERIMENTAL

Materials and Methods

Paperboard with a three-layer structure and grammage of $230~g/m^2$ was used as the coating substrate. The thickness, air permeability, and surface roughness of the substrate were $370~\mu m$, 540~mL/min, and $5.1~\mu m$, respectively. The substrate was coated with solutions containing different ratios of biowax-based component (TopScreen BW200, Solenis LLC, USA) and a water-based PLA dispersion (Landy PL-3000, Miyoshi Oil and Fat Co., Ltd., Japan). The PLA grade is for coating applications with the minimum film formation temperature of $20~^{\circ}C$.

Spray coating was performed using a SPALAS coating system (Nanotrons, USA) by applying two layers onto the substrate. The solid content was optimized as high as possible, suitable for all the coating solutions for spray coating. The coating solutions were diluted with water and used at 20% solid content. The target coat weight for each layer was 12.5 g/m^2 , resulting in a total coat weight of 25 g/m^2 . The coated materials were dried in oven at $70 \, ^{\circ}$ C. The coating composition of each test point (TP) is listed in Table 1.

Table 1. Coating Composition Ratios

	TP1	TP2	TP3	TP4	TP5	TP6
Wax	100	80	60	40	20	0
PLA	0	20	40	60	80	100

The grammage, thickness, tensile properties, air permeance, surface roughness, and water absorption (Cobb $_{60}$) of the uncoated and coated materials were measured according to the ISO 534 (2005), ISO 536 (2012), ISO 1924-3 (2005), ISO 5636-3 (2013), ISO 8791-4 (2007), and ISO 535 (2014) standards, respectively. The materials were conditioned at 23 °C and 50% relative humidity before the measurements.

Contact angles were measured with deionized water and 70% ethanol using a Theta Optical tensiometer (Biolin Scientific AB, Gothenburg, Sweden). The contact angles were recorded for 10 min to evaluate the spread or absorption of water drops on the surface. The drop volume was 3 μ L for both water and ethanol. Uncoated and coated materials were conditioned at 23 °C and 50% relative humidity before measurement.

Scanning electron microscope (SEM) images of the samples were captured using a Hitachi SU3500 instrument (Tokyo, Japan) equipped with a secondary electron detector. A sputter coating with gold was used to coat the sides of the samples and an accelerating voltage of 5 kV was applied.

Creasing experiments with 25-mm-wide sample strips were performed using a special laboratory-scale testing device that simulates the operation of an industrial flatbed die cutter. The testing device was described in more detail in a previous study (Tanninen *et al.* 2021). The device was combined with a Shimadzu AGS-X material tester (Electromechanical Tabletop Frame Tester, Shimadzu, Kyoto, Japan) in which the position (\pm 0.1%), speed (\pm 0.1%), and force (\pm 1%) could be adjusted accurately. The dimensions of the creasing tool set, which comprised a form and matrix, were selected based on the thickness of the sample materials according to the recommendations provided by the die-

cutting tool manufacturer. The rule width, groove matrix, and depth during creasing were 0.71 mm (2 pt), 1.2, and 0.4 mm, respectively. Six parallel-creased samples were produced in the sample material along the machine direction (MD) and cross direction (CD). After the formation phase, each crease was manually folded at an angle of 90° and the integrity of the coating layer ensured by applying dyed water on the crease area.

The coated samples were cut into 25 mm-wide strips and sealed on both sides of the sample using an HSB-1 laboratory heat sealer (RDM Test Equipment Ltd, Hertfordshire, United Kingdom) with 25.4 mm-wide flat heated sealing jaws. The samples were sealed using two different sealing time settings of 1 s or 2 s, sealing pressures of 2 bar or 4 bar (surface pressure of 2.40 and 4.80 N/mm² on the sample), and sealing temperatures of 100, 110, 130, or 160 °C. The sealed samples were peeled using the Shimadzu AGS-X 1 kN tensile tester at a jaw separation speed of 300 mm/min. The maximum force required to separate the samples was recorded.

RESULTS AND DISCUSSION

The thickness, air permeance, roughness, and tensile properties of the uncoated and coated materials were measured. The average thickness of the coated materials was $400\pm3~\mu m$, resulting to average change in thickness with 30 μm . Generally, thickness is an important feature in creasing, as the dimensions of the creasing tools are selected for based on material thickness. In addition, coatings with higher thickness have more tendency to crack in creasing and folding, especially if pigments are used (Panek and Hart 2022). However, since the thickness difference between the coated materials was small as seen in Table 2, this is not expected to have major effect on the creasing process.

The air permeance of the materials was measured to evaluate the coating uniformity and presence of defects in the coating. The wax coating decreased the air permeance of the material compared with that of the uncoated substrate (Table 2). The effect was strengthened by the addition of PLA with a further reduction of the air permeance in the coating containing 20% PLA. Uniform coatings with an air permeance of 0 mL/min were obtained when the amount of PLA in the coating reached or exceeded 40%. The reduction in air permeance within PLA addition indicated that PLA improved the formation of a uniform coating.

Table 2. Air Permeance and Surface Roughness of the Uncoated and Coated Materials

Test Point	Air Permeance	Roughness	
	(mL/min)	(µm)	
Uncoated	540 ± 40	5.1 ± 0.1	
TP1	109 ± 5	4.1 ± 0.1	
TP2	7 ± 3	4.1 ± 0.1	
TP3	0 ± 0	4.5 ± 0.3	
TP4	0 ± 0	4.4 ± 0.2	
TP5	0 ± 0	5.4 ± 0.1	
TP6	0 ± 0	3.5 ± 0.2	

In general, the surface roughness of the materials increased with the addition of PLA, which may be due to different film formation characteristics of PLA. However, the pure PLA coating provided the smoothest surface among all the coated materials. Surface

roughness affects gloss (Samyn *et al.* 2011), which was observed with TP5. TP5 exhibited a more matte characteristic during the visual evaluation of the materials as well as a rougher surface compared to the other coated materials.

The tensile indices of the coated material were lower than that of the uncoated material (Fig. 1). Although PLA coatings have been reported to increase the tensile strength and elongation of materials (Rhim and Kim 2007), the substrate underwent relatively high wetting during the coating process in this study. Wetting and drying may result in reduced tensile strength of the coated materials. In addition, the possible absorption of the coating solution into the substrate may cause a similar effect (Rhim *et al.* 2009). The elongation of the coated materials increased with the addition of PLA, especially along the CD. However, the increase in elongation was not linear in TP3 and TP5, in which 40% and 80% PLA were added. The elongation exhibited the smallest increase in TP1, probably because of the brittleness of the wax coating. In general, the elongation behavior of a material is a more critical parameter during converting than its tensile strength (Vishtal and Retulainen 2012).

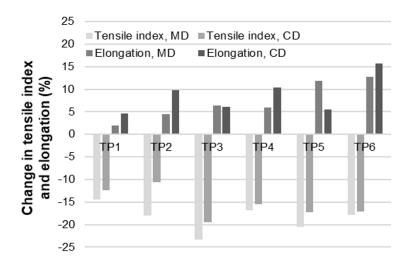


Fig. 1. Tensile index and elongation of the coated materials expressed as changes with respect to the uncoated material. MD and CD refer to the machine and cross directions, respectively.

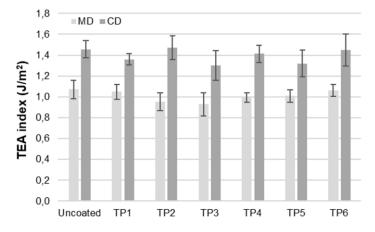


Fig. 2. Changes in the tensile energy absorption (TEA) of the coated materials compared to that of the uncoated material

Figure 3 shows SEM images of the coated surfaces. The SEM images reveal the presence of small cracks in the wax coating (Fig. 3A), which may explain the higher air permeability of the material. The high wetting of the materials during the coating process may have affected crack formation. Crack formation in biowax coating has also been reported in a study by Jo *et al.* (2022), where higher drying temperature as used. The number of cracks decreased as the amount of PLA in the coatings increased. The lighter particles in the SEM images may indicate the presence of wax on the coating surface as waxes tend to migrate to the surface (Rastogi and Samyn 2015) (Figs. 3B, 3C, 3D, and 3E). In addition, changes in the surface in wax-containing coatings have been reported elsewhere (Apicella *et al.* 2022). Despite the notable decreases in air permeability, defects were observed in TP2 and TP3 within the lighter areas. This may indicate that the defects observed in the SEM images were in the top layer or that the addition of PLA led to the formation of more uniform coating layers. Based on visual evaluation, the coating was more uneven for TP3, whereas TP5 (Fig. 3E) had a more matte appearance. The use of PLA (Fig. 3F) resulted in a uniform coating layer.

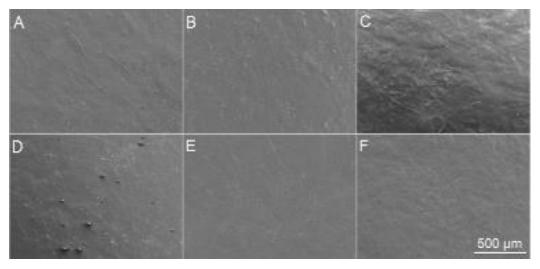


Fig. 3. SEM images of the coated materials: (A) TP1, (B) TP2, (C) TP3, (D) TP4, (E) TP5, and (F) TP6

The water contact angles on the uncoated and coated surfaces were measured to evaluate the effects of the coating and coating composition on the wettability of the surface (Fig. 4). The uncoated surface was more hydrophobic than the coated surfaces, possibly because of the sizing and roughness of the uncoated material. The PLA coating exhibited a notably lower water contact angle. However, even with small amounts of wax in the coating, the surface was hydrophobic, resulting in water contact angles greater than 100°. Increasing the amount of PLA in the coatings increased the wettability of the surfaces. Similar effect has been reported also by Apicella *et al.* (2002). The behavior of the contact angle over time was similar for all the wax-containing coatings.

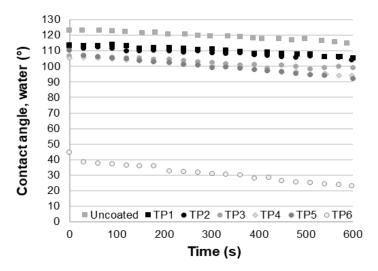


Fig. 4. Contact angle of water on uncoated and coated surfaces

The contact angles of ethanol (70%) were measured for 10 s on uncoated and coated surfaces to observe if defects were present in the coating layers (Fig. 5). On the uncoated surface, the drop was immediately absorbed and penetrated the substrate, and the contact angle could not be recorded. On the wax-coated surface, the drop was absorbed and spread completely within 5 s, and the water contact angle decreased slightly over 10 min (Fig. 4). The absorption of ethanol can be ascribed to small cracks in the wax coating and the lower surface tension of ethanol compared to that of water. On the PLA coating, ethanol was absorbed and spread on the surface within 3 s. Ethanol did not penetrate the wax- and PLA-containing coatings.

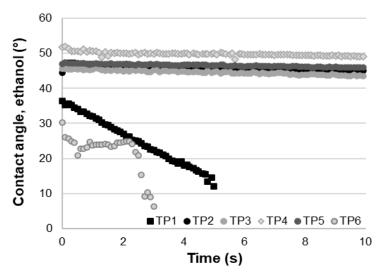


Fig. 5. Contact angle of 70% ethanol on coated surfaces

Water absorption was almost nonexistent (0 g/m²) in the coating with the highest amount of wax (Fig. 6) despite the defects in the coating layer observed in the SEM images. The multilayered structure and hydrophobicity of the coating may have hindered water penetration. As the PLA content increased to 40% and beyond, water absorption started to

increase. However, surfaces of the wax containing coatings were found hydrophobic. Increased water absorption for these may be due to changes in the composition of the coating (Mesic *et al.* 2010; Samyn *et al.* 2010), and the decreased amount of wax may partially explain the increased water absorption at higher PLA contents. The coating with 40% PLA (TP3) dispersion was uneven and patchy. In addition, the increase in water absorption on this surface was similar to that of the pure PLA coating. This may indicate an uneven distribution of coating components on the surface, which resulted in higher water absorption due to PLA. Based on the contact angle measurement results, the PLA-coated surface was considered hydrophilic. The highest water absorption occurred in the PLA-coated material, which also exhibited a higher water uptake compared to that of the uncoated material.

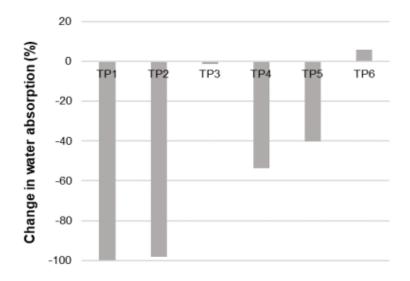


Fig. 6. Change in water absorption of the coated surfaces compared to that of the uncoated surface. The change in water absorption with the percentage of -100 refers to water absorption of 0 g/m^2 .

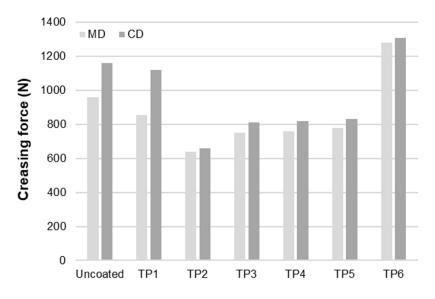


Fig. 7. Maximum creasing force that could be applied without coating layer failure

The maximum creasing force for the coated samples was determined by increasing the force in increments of 20 N until the force could not be endured by the coating layer. The maximum force can be used to assess the suitability of the sample materials for industrial production. A small maximum force hinders reliable industrial production because of the reduced or even nonexistent force adjustment range.

The creased and folded sample strips were visually evaluated, and the breakage of the coating layer confirmed by applying dyed water to the crease area. The highest creasing forces that could be applied without inducing failure in the coating layer are plotted in Fig. 7.

The results show that the samples could withstand the stresses of converting process with suitable magnitudes of the creasing forces (Tanninen *et al.* 2021) in the coating layers for industrial converting in these respects. It should be noted that the force measurement results were slightly higher than the maximum forces used in the experimental setup of an earlier study on the creasing of laminated paperboard (Beex and Peerlings 2009). To provide a safety margin for material quality variations during processing, the optimal creasing force should be lower than the experimentally determined maximum values. The optimal creasing force for the entire sample batch of 25 mm-wide sample strips was determined to be 500 N. A substantially higher force could be applied to samples TP1 and TP6 without concern. Because the same optimal creasing force value was found for all the samples, they could be converted using the same process parameters in industrial die cutting in, for example, successive batches.

The durability of the coatings during folding was evaluated from the SEM images (Fig. 8). The creased and folded areas in the wax coating and the coating with the lowest PLA content were damaged (Figs. 8A and 8B) with severe cracking and breaking in the coating layer, possibly indicating weak adhesion between the substrate and coating layer. The decreased number of cracks (Figs. 8C and 8D) after the addition of PLA can be ascribed to the flexibility of the PLA film. Minor defects were observed in the coating with the lowest amount of wax (Fig. 8E) and the pure PLA coating (Fig. 8F). These defects were likely to have formed during the coating or drying phases. Although the creasing force was set near the failure limit of the coating layer, damage caused by the creasing tool was not detected in some cases.

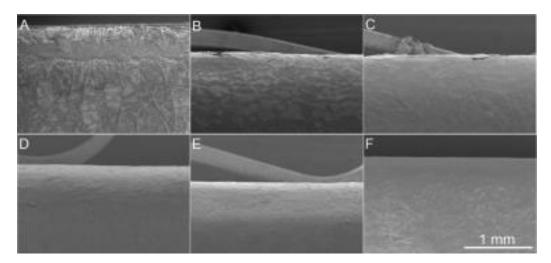


Fig. 8. SEM images of the creased and folded samples: (A) TP1, (B) TP2, (C) TP3, (D) TP4, (E) TP5, and (F) TP6. The samples were creased with the maximum forces as shown in Fig. 7.

Figure 9 shows the maximum force required to separate the sealed materials. The wax coating was not sealed in the tested temperature range (Fig. 9A), while the addition of a small amount of PLA had only a minor effect at higher pressures (Fig. 9B). These results could be explained by the lower sealing properties of the waxes (Yadav *et al.* 2024). In contrast, the addition of PLA increased the sealing properties and seal strengths of the materials. The sealing temperature had a greater effect on the seal strengths of TP3 and TP4, in which the coatings contained 40% and 60% PLA, respectively.

The highest seal strengths were recorded for TP6, which consisted only of PLA. The pressure did not seem to have a notable effect on the seal strength of PLA at longer dwell times. In addition to the heat sealing parameters, the sealing properties are affected by, for example, the surface roughness and surface energy (Ilhan *et al.* 2021). TP5 exhibited the highest surface roughness and a high seal strength over a wide sealing temperature range, even for a short sealing time of 1 s. Wide sealing temperature ranges with short sealing times are important in food packaging applications and production lines (Ilhan *et al.* 2021). A longer sealing time resulted in only a minor increase in seal strength; however, increased seal strength was noted at low temperatures.

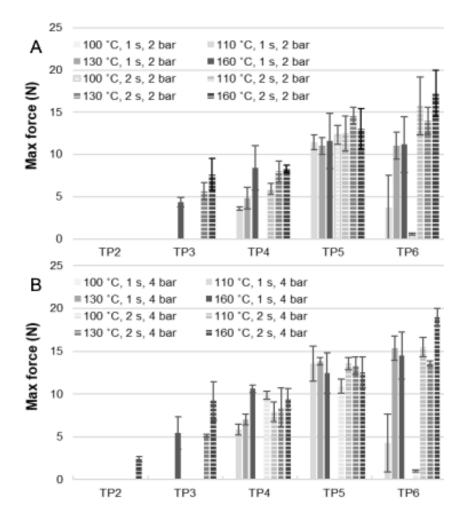


Fig. 9. Seal strengths of the coated materials at different temperatures, the dwell times of 1 s and 2 s, and pressures of (A) 2 bar and (B) 4 bar

CONCLUSIONS

- 1. Adding a small amount of biowax to the coating may improve its water resistance without degrading the convertibility of the material.
- 2. All the samples were found to be sufficiently durable for the converting methods in this study (cutting, creasing, and folding). The addition of polylactic acid (PLA) decreased defect formation in the coating and increased its durability.
- 3. The presence of wax in the coatings increased the hydrophobicity of the surface, whereas increasing the amount of PLA increased water absorption in the coated materials.
- 4. The heat-sealing properties were improved by the addition of PLA. The addition of the smallest amount of wax expanded the sealing temperature range.

ACKNOWLEDGMENTS

This research was partly funded by ECOtronics project (Funder: Business Finland, grant number 1423/31/2019), which is gratefully acknowledged. Teija Laukala is thanked for SEM images. Mahdi Merabtene is thanked for assisting in laboratory work. Stora Enso is acknowledged for providing the substrate.

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Article submitted: July 4, 2024; Peer review completed: August 1, 2024; Revised version received and accepted: October 17, 2024; Published: October 29, 2024. DOI: 10.15376/biores.19.4.9631-9644