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Enhancing the Mechanical Strength and Thermal Stability of Polylactic Acid (PLA) with the Addition of Epoxidized Waste Cooking Oil (EWCO)

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ABSTRACT

Polylactic Acid (PLA) comes from renewable resources, has a reasonable biodegradability rate, and is used in biomedical, food packaging, textiles, and agricultural applications. PLA offers high mechanical strength and the ability to compost, similar to polyethylene terephthalate (PET) and nylon. However, the brittleness of PLA has always limited its usage. Therefore, bio-based plasticizers in the biopolymer matrices can increase flexibility (elasticity), durability, and workability. This study aims to determine the optimal blending ratio for the PLA blended with epoxidized waste cooking oil (EWCO) to enhance the mechanical and thermal properties of PLA/EWCO. The mechanical strength test consists of the hardness test (N/mm^2), flexural strength (MPa), and impact energy (kJ/m^2) adopted to evaluate the plasticizing characteristics. The thermal stability analysis involves glass transition temperature (T_g) (°C), cold-crystallization temperature (T_{cc}) (°C) and melting temperature (T_m) (°C). The blending ratio is 97.5PLA/2.5EWCO, 95PLA/5EWCO, 92.5PLA/7.5EWCO and 90PLA/10EWCO. As a result, 97.5:2.5 of PLA/EWCO reduces intermolecular interactions by stimulating more free volume in biopolymer chains' mobility and

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enhancing the flexibility and elasticity of the PLA blends. Ultimately, the brittleness of PLA decreased with increasing EWCO bio-based plasticizer.

Keywords: Bioplastics, epoxidized waste cooking oil, mechanical strength, Polylactic Acid (PLA), thermal stability

INTRODUCTION

Plastic does not break down very efficiently in natural environments, where it may take up to a thousand years to degrade. A lack of efficient disposal of plastic waste harms the environment, including air, water, and soil pollution. Thus, researchers focus on biodegradable and natural materials since they are concerned about plastic pollution and its environmental effects, especially in oceans and waterways (Haryono et al., 2017). Bio-based plasticizers from epoxidase vegetable oil can replace petroleum-based plasticizers to improve flexibility and stability (Hosney et al., 2018).

However, besides many other usages, vegetable oil is highly demanded for human consumption, which urges researchers to look for better alternatives for bio-based plasticizer production that will not compete with food production. Waste cooking oil (WCO) is considered one of the renewable wastes that can be converted into green products, costing two to three times less than virgin vegetable oil (Benti et al., 2023). It could be an excellent option for producing bio-based plasticizers.

Many excellent benefits are offered by WCO, including the fact that it is low-cost, more accessible for purchase, and environmentally friendly. In addition to recycling WCOs, it also prevents improper waste disposal, which poses a threat to the environment (Suzuki et al., 2018). There are numerous advantages to using epoxidized WCO (EWCO) as a bio-based plasticizer, especially regarding environmental protection. As a result of the epoxidation reaction, carbon-carbon double bonds are converted into oxiranes (epoxides) with the aid of reagents such as air oxidation, hypochlorous acid, hydrogen peroxide, and organic peroxides.

Additional epoxidized vegetable oil as bio-based plasticizers in thermoplastic monomers such as polylactic acid (PLA) are reported to affect the mechanical properties positively (Suzuki et al., 2018). PLA is a thermoplastic monomer made from organic and renewable sources, such as corn starch, vegetable oil, and sugar cane (Pellis et al., 2021). Even though PLA has many advantages, including excellent mechanical strength, transparency, composability, and safety, it also has poor properties, such as brittleness and toughness., which severely limit its applications (Thuy et al., 2018). For applications requiring more flexibility, the plasticity of (PLA) must be improved (Tan et al., 2019), especially in the blending formulation.

According to published research in Table 1, the studies proved that 1-10% of epoxidized vegetable oil (EVO) was confirmed to impact the PLA polymer significantly regarding thermal and mechanical properties (Garcia-Garcia et al., 2020). The proper selection of epoxidized vegetable oil as a bio-based plasticizer for PLA needs to be evaluated on a case-by-case basis as it highly depends on the compatibility of different epoxidized vegetable oil with PLA, and the effect of the bio-based plasticizers percentage on the thermal and mechanical properties of the produced PLA.

Although many studies have explored the effects of different blending ratios on flexural strength, impact energy, and hardness, there is still a potential research gap in determining the ideal blending ratio for maximizing the mechanical strength and thermal stability of the PLA/EWCO simultaneously. Moreover, further exploring how varying processing parameters, such as the blending technique, temperature, and sequence of addition, can influence the final properties of PLA/EWCO blends may help enhance our understanding of attaining the desired mechanical and thermal properties in these bio-based composite materials.

As a result of the numerous tests of PLA /EWCO on mechanical properties and thermal properties above, several advantages, disadvantages, and optimal blending ratios have been identified based on the ratio value set on PLA/EWCO based on the comparison between those two properties. Based on the research done by the researcher before, it was found that the trend of the properties and enhancement is similar based on mechanical and thermal properties.

It is crucial to optimize the optimal blending formulation ratio using the Analytic Hierarchy Process (AHP). EWCO is a renewable bio-based plasticizer that can be produced from waste cooking oil. It is a non-toxic and biodegradable material that has been shown to improve the flexibility and toughness of PLA (Siracusa et al., 2008).

Compared to pure PLA, a blend of PLA/EWCO may offer some advantages. Due to its flexibility and toughness, it is more resistant to breaking than other materials. Moreover, it is also biodegradable and compostable, making it a more sustainable material (Lasprilla et al., 2012).

According to previous studies in Table 1, the optimal composition of EWCO can significantly enhance the mechanical and thermal properties of bio-based plasticizers. As Garcia-Garcia et al. (2020) reported, the impact energy increased with a higher bio-based plasticizer content of up to 2.5% of EKO. All these results clearly show that PLA-2.5% EKO bio-based plasticizer has good mechanical properties.

Thermal properties result from the study by Garcia-Garcia et al. (2020), incorporating 2.5% of EKO as a bio-based plasticizer into PLA produced a saturation of bio-based plasticizers in the polymeric matrix and only a modest increase of crystal orientation. However, in the study by Thuy et al. (2018), with the same amount of 5% addition of EeRSO into PLA, as the glass transition temperature decreased, the crystal content and melting temperature decreased, and the initial decomposition temperature increased, those properties improved. Also, a study by Chieng et al. (2014) claimed that 5 wt% epoxidized vegetable oil (EVO) produced better results in thermal properties than PLA.

Table 1
Effect of different blending formulations of PLA and epoxidized vegetable oils on the mechanical and thermal properties

Vegetable Oils	Blending Formulation Ratio		Thermal properties			Mechanical properties	Authors
	Polymer	Plasticizer	Glass Transition Temperature (°C)	Cold-crystallization temperature (°C)	Melting temperature (°C)	Impact Energy (kJ/m²)	
Epoxidized Keranja Oil (EKO)	PLA (100%)	0	61.2	98.8	173	34-38	Garcia-Garcia et al., 2020
	PLA (99%)	EKO (1%)	61.1	93.1	171.8	35-39	
	PLA (97.5%)	EKO (2.5%)	60.4	91.3	171.4	37-41	
	PLA (95%)	EKO (5%)	58.3	90.4	171	49-53	
	PLA (90%)	EKO (10%)	55	90.6	171.3	47-51	
Epoxidized Rubber Seed Oil (EeRSO)	PLA (100%)	0	65	115.5	167.5	0.0009-0.0095	Thuy et al., 2018
	PLA (95%)	EeRSO (5%)	60	112.5	165.5	0.0180-0.0184	
Epoxidized Palm Oil (EPO)	PLA (100%)	0	62.85	124.11	149.79	-	Chieng et al., 2014
	PLA (100%)	EPO (5%)	60.12	114.16	147.62	-	
	PLA (100%)	EPO (7%)	60.79	108.97	146.97	-	

MATERIALS AND METHODS

Bio-based Plasticizers Preparation

PLA 3001D poly (lactic acid) (270,000 g mol^{-1}) in the form of pellets was supplied by NatureWorksTM LLC (Omar et al., 2023). The PLA was first dried at 50°C for 2 hours (Giita Silverajah et al., 2012) to prevent the hydrolysis of PLA before being used in blend preparation. For the epoxidized process, 88% of Formic Acid, HCOOH, and 30% of Hydrogen Peroxide H_2O_2 were obtained from Systerm Chemicals Company in Malaysia. PLA and EWCO were manually mixed until they were well blended in the blending formulation ratio 97.5PLA/2.5EWCO (Garcia-Garcia et al., 2020), 95PLA/5EWCO (Thuy et al., 2018), 92.5PLA/7.5EWCO (Chieng et al., 2014) and 90PLA/10EWCO (Garcia-Garcia et al., 2020), as indicated in Table 2 and Figure 1. PLA can only accommodate about 10% plasticizer due to the potential migration of the plasticizer and the resulting negative impact on its mechanical properties (Marturano et al., 2023).

EWCO was prepared in a 3-necked round bottom flask with 40 g of WCO and 3.7 g of HCOOH stirred at 30°C for 30 minutes. Following that, 32 g of H_2O_2 was dropped into the flask within 30 minutes, and the solution was stirred using a magnetic stirrer for about 7 hours (Cai et al., 2020). EWCO is then washed and rinsed with distilled water.

EWCO was dried at ambient temperature for four hours, while pure PLA was dried at 80°C for four hours (Thuy & Lan, 2021). As hydrolysis produces large amounts of degradation products from monosaccharides, PLA was preheated before blending to prevent hydrolysis (Garcia-Garcia et al., 2020). The percentages in Table 2 indicate how PLA and EWCO were manually mixed. Once mixed, the mixtures were extruded in a twin-screw extruder at temperatures ranging from 165°C to 180°C. After extruding, samples were collected and crushed to produce pellets. As shown in Figure 1, the PLA blends were placed in a rectangular mold, which was then heated at 220°C for 30 seconds (Omar et al., 2023).

Table 2
Blending formulation ratio of PLA/ EWCO

Polymer (PLA %)	Plasticizer (EWCO %)
100	0
97.5	2.5
95	5
92.5	7.5
90	10

Source: Omar et al., 2023

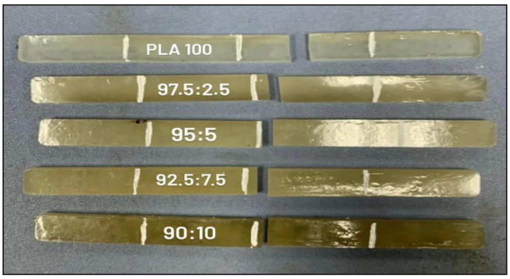


Figure 1. PLA/EWCO

Experimental Mechanical Properties

Rockwell Hardness

According to ASTM E18-22, the Rockwell hardness test can determine a material's hardness. The material sample is a standard size of 6.4 mm (0.25 inch) thickness while the indenter is ball type. The sample types of PLA blended with EWCO were measured five times according to the blending formulation ratio, and the results were analyzed. Five measurements were replicated for each formulation of PLA/EWCO.

Flexural Strength

According to the ASTM D790 standard for plastics, flexural tests were conducted per the standard to determine flexural or bending properties. The size of the specimen is a cross-section of 3.2 mm x 12.7 mm and a length of 127 mm, which is suitable for plastic molding in a universal flexural test machine IBERTEST ELIB 30 (S.A.E. Ibertest, Madrid, Spain), using an operating mode in which it was subjected to three-point bending, as per the operation mode. Each sample related to the PLA blending formulation ratio mixed with EWCO was tested at least five times.

Impact Test

The Charpy impact test (ASTM D2794) was used in this experiment. The materials were evaluated for their ability to absorb/impact energy during fracture using a Charpy pendulum (6 J) from Metrotech S.A (San Sebastian, Spain), which complied with ISO 197:1993 guidelines (Garcia-Garcia et al., 2020). The size of the specimen is 64 x 12.7 x 3.2 mm. Each sample was drilled with a notch containing a depth of 2 mm and a radius of 0.25 mm in the center. Five samples of PLA/EWCO were tested according to the blending ratio, and the average value was calculated.

Experimental Thermal Properties

The thermal properties of polylactic acid (PLA) were analyzed using differential scanning calorimetry (DSC) after plasticizing with epoxidized waste cooking oil (EWCO). The DSC results for the treated and plasticized PLA samples were obtained by heating them in a nitrogen atmosphere at a flow rate of 50 mL/min^{-1}, from 30°C to 700°C under thermogravimetric analysis (TGA) dynamic mode at a heating rate of 10°C/min^{-1}. The plasticizers decreased the PLA's glass transition temperature (T_g), and the degree of crystallinity (Xc) was calculated using the melting and cold crystallization enthalpy from the DSC data.

The analysis conducted a DSC test under a nitrogen atmosphere using a Mettler-Toledo 821 calorimeter (Schwerzenbach, Switzerland) (flow rate 66 mL min^{-1}). As a procedure, the test involved two heating stages at 20°C for 10 minutes each, followed by a cooling stage at 20°C for 10 minutes, and a final heating stage at 250°C for 10 minutes. The sample weight ranged from 5 mg to 20 mg, and the second heating scan aimed to determine the glass transition temperature (T_g), crystallization temperature (T_{cc}), and melting temperature (T_m) of the sample.

Optimization Process

The Analytic Hierarchy Process (AHP) has been used to optimize the best percentage value. This model-based verification AHP optimization aims to identify the optimal blend PLA/EWCO to achieve mechanical properties (Flexural, tensile, and impact energy) and thermal stability (glass transition temperature, cold-crystalization temperature, and melting temperature). A series of pairwise comparisons may produce some inconsistencies.

The Consistency Ratio (CR) is an essential part of the hierarchical framework of AHP since it can determine the consistency of pairwise comparison judgments. For each of the criteria, all comparisons between criteria and alternatives were analyzed for consistency (Razak et al., 2022). The overall AHP analysis has been presented in Figure 2. The reason for adopting AHP is that AHP provides reciprocal pairwise comparisons and has a multi-level hierarchical structure analysis to propose the best alternatives from a discrete set of feasible alternatives

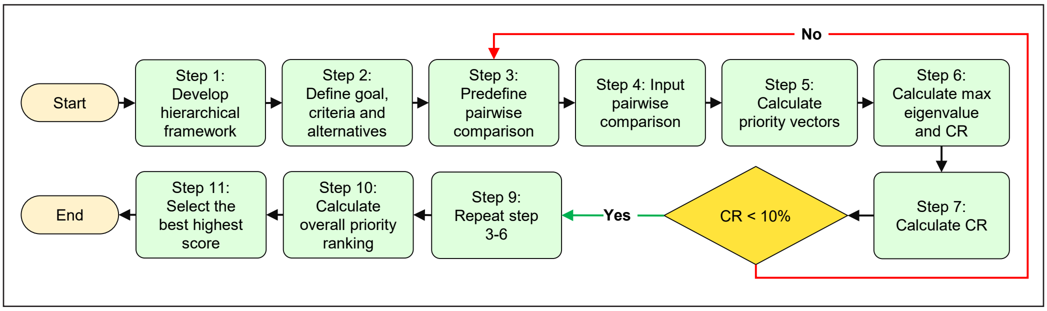


Figure 2. AHP method

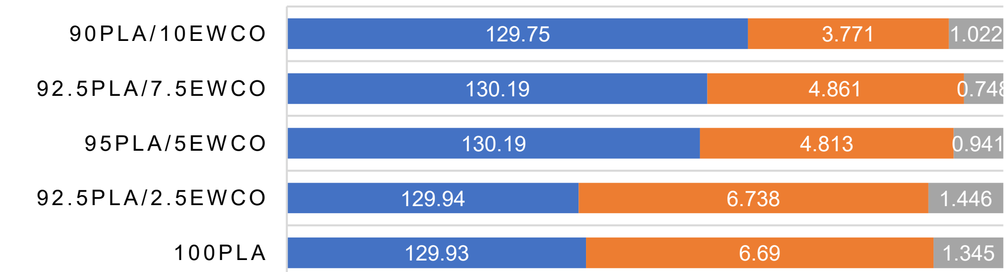
RESULTS AND DISCUSSION

Effect on Mechanical Strength

Figure 3 shows the results on the mechanical properties of the PLA/EWCO plasticizers.

According to the process, PLA becomes less rigid when plasticized with EWCO because epoxidized palm oil reduces tensile strength. There is a possibility that the plasticizer interacts with the PLA matrix, decreasing the intermolecular forces and increasing the mobility of the polymer chains, which results in a decrease in rigidity for the material.

A hardness test can be used to evaluate a material's properties, such as strength, elasticity, and wear resistance, and therefore aid in selecting the appropriate material or treatment for other applications. PLA caused an increase in Rockwell hardness before it became static at 5 EWCO% to 7.5 EWCO%. However, a further increase to 10 EWCO% has caused a reduction in Rockwell hardness. Incorporating EWCO plasticizer into PLA makes the material more flexible and less indentation-resistant than pure PLA. However, extensive addition of EWCO will lead to a more brittle product, as the higher the EWCO is added, the more complex the material becomes.



	100PLA	92.5PLA/2.5 EWCO	95PLA/5 EWCO	92.5PLA/7.5 EWCO	90PLA/10 EWCO
Hardness Test (N/mm²)	129.93	129.94	130.19	130.19	129.75
Flexural test (MPa)	6.69	6.738	4.813	4.861	3.771
Impact energy (kJ/m²)	1.345	1.446	0.941	0.748	1.022

Figure 3. Mechanical properties of PLA/EWCO

According to ASTM D790, the flexural test shows that the more EWCO is added to the PLA, the more the load data increases until the specimen breaks (Rostamiyan et al., 2015). As a result, the strength of the material decreases when EWCO is added, resulting in a weak material when the ratio of EWCO and PLA is 92.5:7.5. Increases in flexural strength can result in higher hardness values, indicating that the material is more resilient and can maintain its shape under load. In contrast, decreased flexural properties can reduce hardness, make it more brittle, and indicate that the material is more susceptible to external forces causing deformation.

The impact energy of PLA/EWCO blends is higher than that of pure PLA. In other words, PLA/EWCO resists sudden breakage under an impact load. Figure 3, results on mechanical properties, shows that the force value decreases when more EWCO values are added to the PLA. This result is similar to Thuy et al. (2018). By adding more EWCO, the PLA will become soft and weak. The lower load transfer capacity of the material is also the reason for the reduced impact strength of the composites during the test (Girimurugan et al., 2020).

The results of two out of three tests in Figure 3 indicated that 97.5PLA/2.5EWCO was the best based on mechanical properties (flexural and impact energy). As a result, the hardness value results of 97.5PLA/2.5EWCO can be considered optimal. Based on the results demonstrated in Figure 3, 97.5PLA/2.5EWCO has the best mechanical strength, such as optimal hardness, higher flexibility, and higher energy impact.

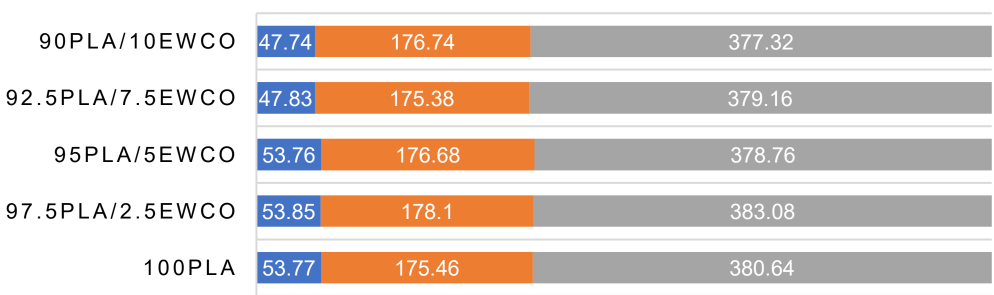
Effect on Thermal Stability

In addition to providing valuable information regarding the thermal stability of materials, DSC can be used to determine their thermal decomposition and degradation behavior by measuring various parameters related to their thermal behavior (Liu et al., 2013). Information such as this is essential for assessing the safety of handling, processing, and storing materials, especially in chemical manufacturing, pharmaceutical manufacturing, and polymer manufacturing.

Thermal stability is significant for this study because the PLA may decompose when heated due to high temperatures. In addition, the time and temperature at which physical changes occur when PLA is heated or cooled when blended with EWCO. According to the data obtained from the DSC Thermal Analysis test and analyzed, Figure 4 summarizes the test results. Adding EWCO to PLA does not result in significant differences from pure PLA according to the ratio set. However, small changes in value can have a significant impact, even if the difference in weight is only minimal.

Heat is applied to a sample at a constant rate, and heat flow is measured to maintain a constant temperature difference between the sample and the reference material. As a result of the DSC curve, information can be obtained about the thermal transitions of the material, including their peak temperatures and enthalpy changes. A DSC test is usually conducted in three stages, such as heating, cooling, and reheating, to determine the thermal properties of a polymer PLA. During the first heating stage, the thermal history of the sample is erased, while the cooling and reheating stages provide information regarding the cold-crystallization and melting of the polymer. Reheating determines the glass transition temperature (T_g) while cooling and reheating determine the cold-crystallization temperature (T_{cc}) and melting temperature (T_m).

As shown in Figure 4, The glass transition temperature (T_g) of pure PLA is around 60°C, but when EWCO plasticizer is added, it lowers the T_g of PLA to below 53.77°C,



	100PLA	97.5PLA/2.5 EWCO	95PLA/5 EWCO	92.5PLA/7.5 EWCO	90PLA/10 EWCO
Glass transition temperature (°C)	53.77	53.85	53.76	47.83	47.74
Cold-Crystallization temperature (°C)	175.46	178.1	176.68	175.38	176.74
Melting temperature (°C)	380.64	383.08	378.76	379.16	377.32

Figure 4. Thermal properties of PLA/EWCO

making it more flexible and less rigid (Turco et al., 2019). However, adding too much EWCO can produce a weak and soft material. PLA's cold-crystallization temperature (T_{cc}) is between 170°C and 180°C, but when EWCO plasticizer is added, it disrupts the crystal structure, making it harder for the material to crystallize. The T_{cc} increases with EWCO but decreases when more than 5% is added. PLA's melting temperature (T_m) is around 290°C–380°C (Carrasco et al.). However, when EWCO plasticizer is added, it decreases, and the melting temperature value becomes unstable when the addition of EWCO exceeds 5%. This instability in Tm may be caused by changes in the PLA crystal structure brought about by EWCO plasticizers.

During crystallization, the temperature affected the degree of crystallinity of the polymer, which, as a result, affected the melting temperature (Scoti et al., 2024). As the degree of crystallinity in polymers increases, various material properties, such as hardness, strength, and barrier properties, improve. Techniques such as Differential Scanning Calorimetry (DSC) determine the degree of crystallinity. Due to thermodynamic regulation, the crystallization temperature is always lower than the temperature at which the melting occurs. As a result of their chemical modifications and crystallization abilities, 2.5 EWCO can alter the crystallization and melting behavior of polymers, impacting their thermal properties. Based on the results in Figure 4, 97.5PLA/2.5EWCO has the best thermal stability, such as higher glass transition temperature, cold crystallization temperature, and melting temperature.

Figure 5a) PLA/EWCO (97.5:2.5) indicates the DSC curve suggests a shift in the baseline around 53.85°C, which means the occurrence of a "glass transition." In addition, a chemical reaction of a mix of reagents generates an exothermic peak of around 178.1°C, indicating an exothermic reaction caused by crystallization. Observing an endothermic peak around 383.08°C indicates an endothermic reaction by melting. It can be seen in Figure 4 that no significant differences exist between graphs a,b,c, and d. in Figure 5.

Optimum Blending Formulation of EWCO for Enhancing Thermal and Mechanical Properties of PLA

The mechanical and thermal properties results have been optimized using the Analytic Hierarchy Process (AHP) to get an average calculated value of PLA/EWCO. In the AHP, model-based formulations were evaluated for their ability to achieve the trade-off between mechanical strength (hardness, flexural strength, impact energy) and thermal stability (glass transition temperature, cold crystallization temperature, melting temperature).

Figure 6 shows that flexural strength is the high-priority attribute and characteristic for selecting the optimal blending of PLA/EWCO, followed by hardness and impact, while for thermal stability, glass transition temperature is the main factor, followed by cold-crystallization temperature and melting temperature. According to Figure 7,

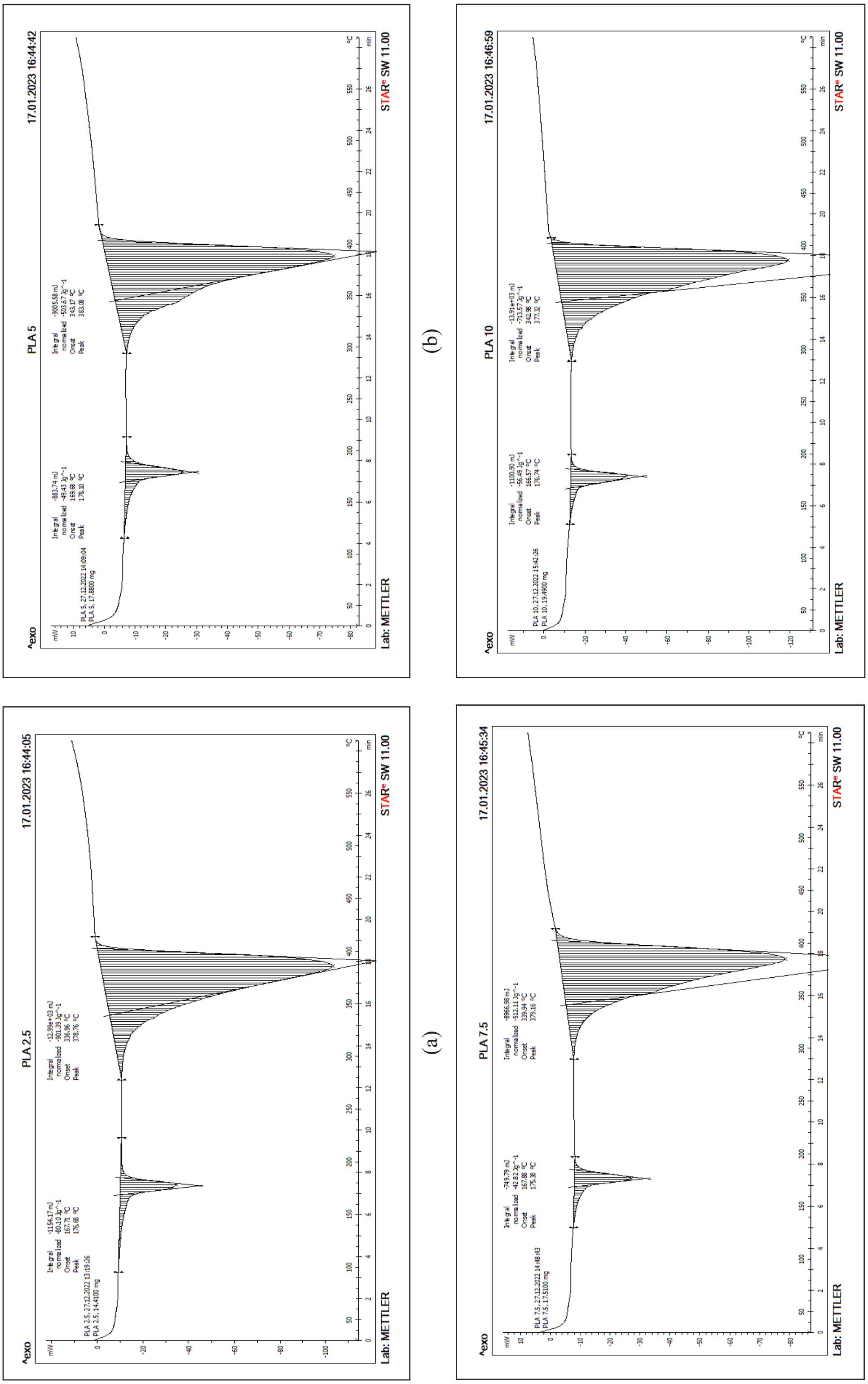


Figure 5. (a) PLA/EWCO (97.5:2.5); (b) PLA/EWCO (95:5); (c) PLA/EWCO (92.5:7.5); and (d) PLA/EWCO (90:10)

100PLA has the highest percentage followed by 97.5PLA/2.5EWCO, 95.5PLA/5EWCO, 92.5PLA/7.5EWCO and 90PLA/10EWCO. As shown in Figure 7, AHP has generated a combination of weight criteria based on mechanical and thermal properties.

Blending the formulation of PLA/EWCO with the ratio of 97.5PLA/2.5EWCO resulted in the highest mechanical and thermal properties. When PLA is combined with the EWCO plasticizer, it disrupts the crystal structure of PLA, hindering crystallization and promoting an amorphous structure. This disruption may occur due to the bulky epoxidized oil molecules interfering with the close packing of PLA chains, thus inhibiting the formation of highly ordered crystals. The addition of 2.5% EWCO enhances the interfacial interaction between PLA and EWCO, thereby enhancing mechanical strength through Van der Waals force and acid-base interactions (Sumesh et al., 2022) and optimizing the properties of PLA in terms of plasticity, mechanical properties, and thermal stability. This enhanced performance is attributed to the plasticizing effect of EWCO, which reduces intermolecular forces between PLA chains, allowing increased mobility and deformation of the chains under stress.

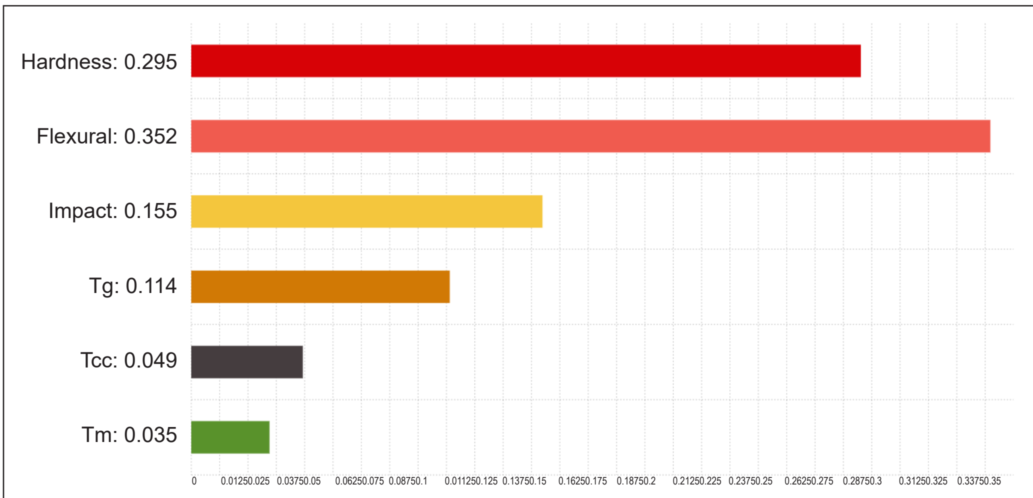


Figure 6. Weight criteria for mechanical and thermal properties

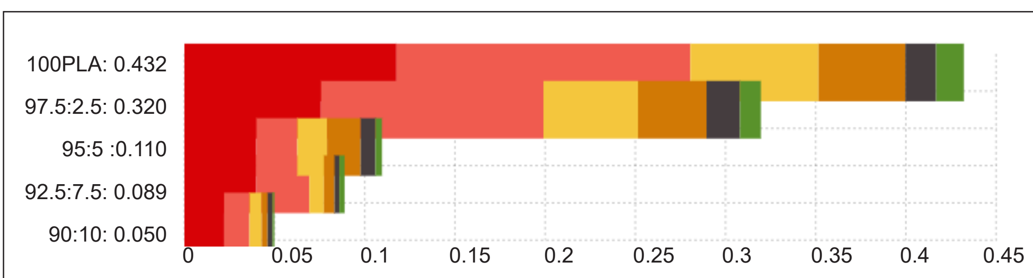


Figure 7. Scored alternatives for optimal blending formulation PLA/EWCObio-based plasticizers

CONCLUSION

This paper has the potential of EWCO in enhancing PLA's mechanical properties and thermal stability. The results indicate that 97.5:2.5 of PLA/EWCO reduces intermolecular interactions by stimulating more free volume in biopolymer chains' mobility and enhancing flexibility in mechanical properties. Meanwhile, the thermal stability increased with increasing bio-based plasticizers up to 2.5% of EWCO based on the AHP analysis that has been done on all properties, from glass transition temperature to cold crystallization temperature and melting temperature.

CREDIT AUTHORSHIP CONTRIBUTION STATEMENT

Nur Batrisyia Norhazlin: Writing the original draft, methodology, software, and investigation; Nurul Hanim Razak: Conceptualization, methodology, validation, proofreading; Anis Ainaa Omar: Data curation, formal analysis; Mohd Hafidzal Mohd Hanafi: Methodology, writing-review and editing; Asmah Mat Desa: Investigation and proofreading.

DECLARATION OF COMPETING INTEREST

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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