Preparation and enhanced mechanical properties of epoxy resin modified with pyrolysis bio-oil

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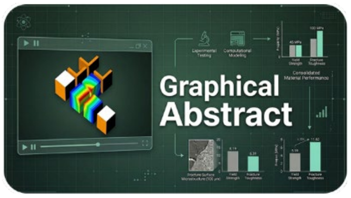

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ABSTRACT. This study investigates the modification of epoxy resins with pyrolysis bio-oils derived from plant waste, aiming to enhance mechanical performance. A method for epoxy resin modification using bio-oil is proposed, including techniques for bio-oil pretreatment, purification, and incorporation into epoxy resin. Three pyrolysis bio-oils (birch, sunflower, and a mixed softwood and hardwood feedstock) across different concentrations were used as a modifier. Mechanical properties were evaluated under tension, compression, and three-point bending loading.

Mechanical testing revealed that epoxy resin modified with the bio-oil at an optimal concentration of 12.5 phr produced polymer system with strength characteristics comparable to epoxy resins modified with an industrial plasticizer, while demonstrating superior compressive strength properties. Combined modification with bio-oil and industrial plasticizer dibutyl phthalate resulted in enhanced both deformation and ultimate stress levels for all types of loading considered.

The bio-oil derived from the mixed wood feedstock can effectively replace the dibutyl phthalate in terms of mechanical performance while providing additional benefits in environmental sustainability and cost efficiency.

KEYWORDS. Epoxy resin, Modification, Bio-oil, Strength, Plasticity.

INTRODUCTION

Epoxy resins are among the most demanded polymer materials in modern industry due to their valuable properties, in particular chemical resistance, excellent adhesion, low curing shrinkage, and high dielectric performance. However, epoxy resins have certain disadvantages, including low deformation characteristics and impact strength. These limitations can be addressed through innovative epoxy resin modification techniques [1–3]. The high reactivity of epoxy groups enables modification of epoxy resins with both inert and reactive additives to produce materials with required specified properties [4].

Modification of epoxy resins is employed to address the following objectives: improving technical and operational characteristics; increasing the technological effectiveness of material, as well as the production process; reducing cost and ensuring environmental safety. The issues of economic efficiency and environmental safety can be jointly resolved by using agricultural waste derivatives as modifiers for epoxy resins. This approach reduces the material cost by decreasing expenses of initial components, as well as minimizes environmental impacts associated with waste disposal. On the other hand, the growing demand for eco-friendly materials, depletion of hydrocarbon resources and tightening environmental regulations also stimulate the development of bio-based polymers. Particularly promising is the use of renewable resources and organic waste as additives in polymer systems [5–10]. Extensive research validates the effectiveness of sustainable modifiers derived from organic waste streams (nut shells, rice husks, date seeds), lignocellulosic fibers (bamboo, hemp, pineapple), food industry waste for enhancing epoxy polymer properties, including improved fracture resistance characteristics [11–15]. As demonstrated in [16] the use of synthetic silicates (wollastonite, diopside) derived from rice husk ash, activated rice husk ash, and epoxidized plant oils enhances both the mechanical properties and tribological properties of coatings.

Plant-based industrial waste from the agricultural complex, forestry, and related industries constitutes a significant portion of global industrial waste. Pyrolysis represents one of the most promising methods for processing these types of waste. Pyrolysis conducted at 450–550°C under an oxygen-free environment induces thermal degradation of biomass-derived organic components, yielding gaseous, liquid, and solid products simultaneously. Biochar, a solid pyrolysis product, exhibits a high porosity and extensive specific surface area, positioning it as a promising filler in polymer composites—an economical alternative to expensive nanomaterials such as carbon nanotubes and graphene. Incorporating of biochar into epoxy matrices improves mechanical and electrical properties and provides additional functionality including antimicrobial properties and flame resistance [7,8,17].

The liquid pyrolysis products (bio-oil) exhibit an extremely complex composition, comprising over 255 organic compounds (acids, phenols, alcohols, sugars, esters, etc.) [18]. The compound distribution shows strong dependence on the biomass source (wood, agricultural waste, peat) and specific pyrolysis parameters. Bio-oil may serve as effective reactive modifiers in epoxy systems, improving their mechanical performance and stress-strain behavior [19,20]. Study [19] revealed that incorporating bio-oil and biochar into epoxy resins enhances mechanical properties, specifically increasing Young's modulus and tensile strength. Furthermore, the optimal content of these additives was determined to maximize both strength and stiffness. In [21] various amounts of hemp bio-oil were used to partially replace the amine-based curing agent in an epoxy resin. The reduction in active crosslink density due to the presence of bio-oil resulted in a more flexible structure, which caused a decrease in Martens hardness and indentation modulus, but improved damping and wear resistance as the amount of bio-oil added to the formulation increased. As demonstrated in [22], modifying epoxy resins with wood processing by-products (including bio-oil) improves both ductility and adhesion in epoxy systems. It should be noted that studies utilizing plant resources typically focus on local feedstocks, which limits the generalizability of results. The properties of resulting materials can vary significantly depending on the specific source material used. Consequently, new type of plant waste used for bio-oil production requires investigation of its effects on the properties of modified epoxy resin.

This study investigates the influence of bio-oil type and concentration on the strength and deformation properties of epoxy resins. The study involved a series of mechanical tests to evaluate the effect of modifiers on parameters including deformation and strength under different loading conditions. The findings reveal new opportunities for developing high-performance biopolymers with specified strength and deformation properties.

MATERIALS AND METHODS

Materials

Bio-oils were produced from three distinct feedstocks - birch, sunflower, and a mixed softwood/hardwood biomass - via slow pyrolysis at 550 °C under atmospheric pressure. The process, regulated by a data acquisition system, featured a 3-hour biomass residence time to ensure complete conversion. The resulting bio-oils are hereafter referred to as BO_B (birch-derived), BO_S (sunflower-derived), and BO_M (mixed wood-derived).

The chemical composition of the purified BO_M was analyzed by gas chromatography with mass spectrometric detection (GC-MS). The GC-MS analysis was performed on a GCMS-QP2010 Ultra chromatograph-mass spectrometer (Shimadzu, Japan) equipped with a BP-1 capillary column. A total of 247 organic compounds were detected, of which 49% were identified. The main identified classes were phenolic compounds (31.5%), ketones (6.5%), carboxylic acids (6.4%), esters (4.0%), and aldehydes (0.6%). The full list of compounds is given in [23].

The bio-oils were used to modify a base polymer system composed of ED-20 epoxy resin cured with polyethylene polyamine (PEPA). The baseline formulation of this system consisted of 100 parts per hundred resin (phr) of ED-20 and 12 phr of PEPA. The optimal PEPA content (12 phr) was determined experimentally for the neat epoxy resin [24]. The same hardener content was used for all bio-oil modified formulations. All blends were visually homogeneous and showed no phase separation prior to curing.

The bio-oils were incorporated at different loadings: BO_B and BO_S at 12.5 phr, and BO_M across a range of 5-25 phr. For comparison, a baseline composition modified with dibutyl phthalate (DBT) was used. The complete formulations of all studied polymer systems are summarized in Tab. 1.

	Epoxy resin (E), phr	Hardener (P), phr	Bio-oil	DBT, phr
EP	100	12	-	-
EP/DBT_12.5	100	12	-	12.5
EP/DBT_25	100	12	-	25
EP/BO_B_12.5	100	12	12.5 phr BO_B	-
EP/BO_S_12.5	100	12	12.5 phr BO_S	-
EP/BO_M_5	100	12	5 phr BO_M	-
EP/BO_M_12.5	100	12	12.5 phr BO_M	-
EP/BO_M_12.5/DBT_12.5	100	12	12.5 phr BO_M	12.5
EP/BO_M_20	100	12	20 phr BO_M	-
EP/BO_M_25	100	12	25 phr BO_M	-

Table 1: Formulations of the investigated polymer systems

Preparation of bio-based epoxy systems

Prior to incorporation into epoxy systems, bio-oils require purification to remove solid pyrolysis residues and eliminate water. The water content in bio-oils can reach up to 30%, depending on the moisture content of the initial feedstock. When the water content exceeds 30%, phase separation occurs, resulting in two distinct phases: water-soluble compounds and heavy pyrolysis resins. Therefore, to ensure the bio-oil homogeneity, water removal is essential and can be achieved through one of the following methods:

- Sedimentation;
- Drying in an oven at 100–105°C;
- Distillation, including vacuum distillation;
- Water extraction.

To remove solid particles, it is recommended [10] to dilute bio-oils with monohydric alcohols (in a 1:1 ratio), such as methanol or ethanol. The diluted bio-oil should be vacuum-filtered through filter paper (red ribbon, thickness 5–8 µm, or white ribbon, thickness 8–12 µm). The mixture is then vacuum distilled at 70°C to remove the solvent. Finally, the purified bio-oil should be stored in a refrigerator at 4°C until use.

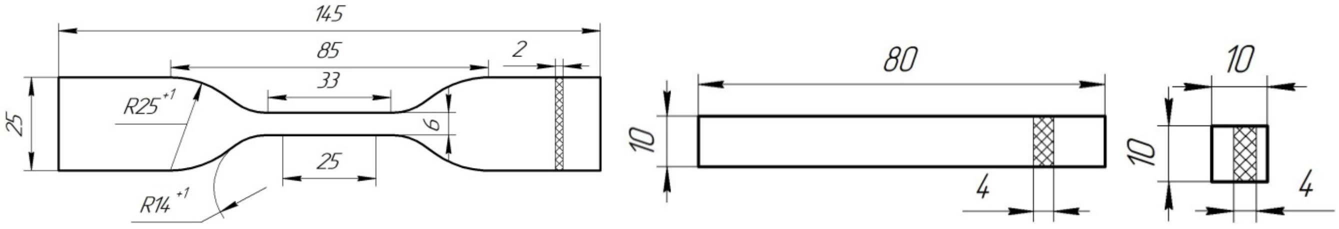
Bio-oils can be introduced into the epoxy system by mechanical mixing, which is one of the simplest methods for modifying epoxy compositions. However, during the modification process, it is crucial to achieve uniform distribution of the modifier throughout the epoxy polymer volume. This can be accomplished by the following methods:

- Mixing the components after pre-heating the bio-oils;
- Mixing the components after pre-heating the bio-oils with acetone pretreatment [10].

In this study, the mixture of ED-20 epoxy resin and purified bio-oils was subjected to acetone pretreatment. As demonstrated in [10], such treatment reduces the viscosity and improves the homogeneity of the mixture, makes the reactive hydroxyl (–OH) groups of the bio-oil more accessible for subsequent reaction with epoxy groups, and ultimately allows a significant increase in the substitution ratio of epoxy resin by bio-oil without substantial deterioration of the mechanical properties of the composition.

Mechanical analysis

The mechanical performance of the modified epoxy systems was evaluated under tension, three-point bending, and compression tests conducted in accordance with ISO 527-2, ISO 178, and ISO 604, respectively. For each composition listed in Tab. 1, five specimens were tested under each loading type. Specimens were tested to failure in tension and bending, while compression tests were carried out until peak load attainment.



a b c

Figure 1: Specimen configurations for: (a) tension, (b) three-point bending, (c) compression testing

Test specimens were fabricated by casting the prepared epoxy systems into silicone molds pre-treated with a release agent. The geometries and dimensions of the test specimens were established according to applicable standards and are shown in Fig. 1. Following demolding, the samples were thermally cured in an oven using the method described in [24]. After curing, the specimens were ground to remove casting defects. Images of the final specimens are presented in Fig. 2.

a b c

Figure 2: Fabricated specimens for: (a) tension, (b) three-point bending, (c) compression testing

Mechanical testing was performed using an LDS-5L universal electro-mechanical testing system (Jinan Liangong Testing Technology Co., Ltd., China).

RESULTS AND DISCUSSION

Experimental results of ultimate strength, including standard deviations, are summarized in Tab. 2. All values are presented as mean ± standard deviation based on five specimens per formulation and loading type. This table covers all investigated compositions, from the neat resin to systems with various bio-oils, dibutyl phthalate, and their combinations.

The data presented serve as a basis for the subsequent detailed analysis of the effects of bio-oil type, its concentration, and combined modification with a commercial plasticizer on the strength properties of epoxy polymers.

	Ultimate strength, MPa		
	tension	bending	compression
EP	69.08 ± 9.48	130.61 ±5.43	114.3 ± 1.37
EP/DBT_12.5	64.33 ± 2.07	112.62 ±6.12	97.41 ± 0.92
EP/DBT_25	39.87 ± 0.53	70.02 ±4.9	59.17 ± 1.29
EP/BO_B_12.5	54.98 ± 7.2	108.38 ± 10.22	116.40 ± 2.56
EP/BO_S_12.5	60.78 ± 8.77	92.6 ± 8.52	108.02 ± 2.42
EP/BO_M_5	61.03 ± 8.51	101.67 ± 11.67	107.33 ± 1.34
EP/BO_M_12.5	61.69 ± 11.81	106.15 ± 4.03	107.52 ± 2.42
EP/BO_M_12.5/DBT_12.5	46.15 ± 0.67	91.55 ± 2.14	68.68 ± 1.37
EP/BO_M_20	57.81 ± 9.09	92.54 ± 9.89	108.82 ± 3.77
EP/BO_M_25	51.02 ± 8.32	77.27 ± 11.41	107.81 ± 2.64

Table 2: Ultimate strength values (mean ± standard deviation) of the epoxy systems modified

Impact of bio-oil source on strength of modified epoxy resins

The influence of the bio-oil feedstock type on the strength properties of the modified compositions is analyzed in this section. Fig. 3 presents the tensile, compressive, and three-point bending ultimate strength (mean values) values of the epoxy systems. For comparison, the ultimate strength values of the baseline system modified with the common plasticizer DBT are included for each loading type. All modified epoxy systems presented in Tab. 2 contain 12.5 phr of the respective modifier.

The data presented in Fig. 3 indicate that plant waste bio-oils provide ultimate strength equivalent to that achieved with DBT modification under tension and three-point bend loading conditions. Specifically, depending on the feedstock type, bio-oil modified epoxy systems show 4–15% lower tensile strength and 3–18% lower bending strength compared to the DBT-modified reference. Compression testing revealed the significant benefit of bio-oil modification, with all bio-oil types demonstrating superior strength compared to the DBT-modified reference system. Namely, this represents a strength enhancement of 11–39% over the DBT-modified benchmark. Analysis of the strength performance under various loading conditions identified BO_M as the most promising modifier among the three bio-oil types for practical applications. Consequently, the subsequent analysis in this work focused on using BO_M to modify the base epoxy system.

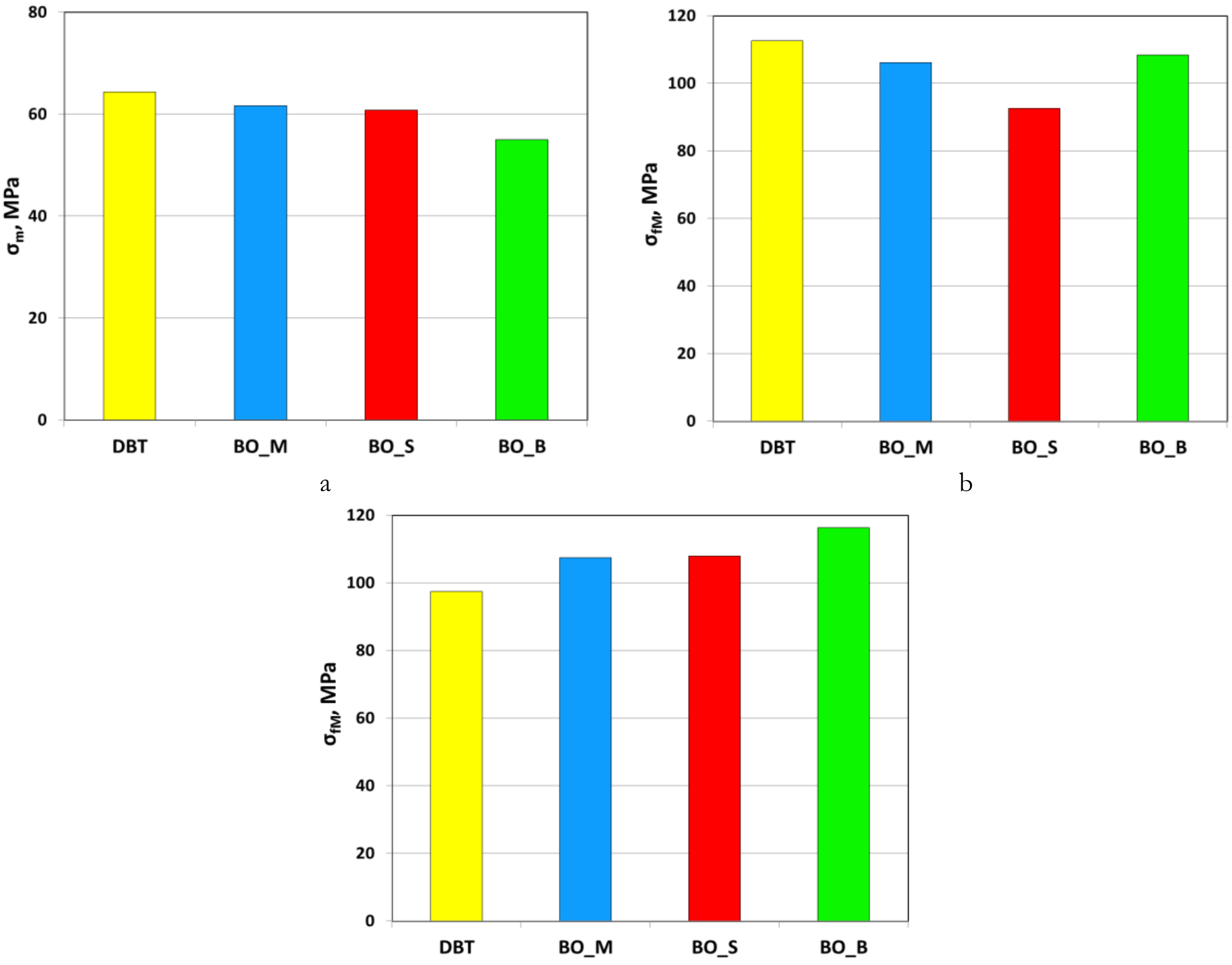


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Figure 3: Ultimate strength values of the epoxy systems modified with 12.5 phr of bio-oils and DBT for (a) tension, (b) three-point bending, (c) compression testing

Stress-strain analysis of modified epoxy systems

Fig. 4 shows representative stress-strain curves of epoxy systems modified with 12.5 phr BO_M, 12.5 phr DBT, 25 phr DBT, and an equal-parts blend of BO_M and DBT (12.5 phr each), compared to the neat epoxy. Each curve corresponds to a single specimen and is shown as a typical example from a set of five tested specimens per formulation. The experimental data indicate that all modifiers reduce the strength of the baseline epoxy system. The epoxy system with 12.5 phr BO_M exhibits mechanical properties close to the 12.5 phr DBT-modified reference system across all loading modes.

It was established that modifying the baseline epoxy system with equal parts of BO_M and DBT (12.5 phr each) enhances the mechanical properties compared to the system modified with DBT alone (25 phr). In other words, replacing half of the DBT mass fraction in the total plasticizer composition with BO_M - while maintaining a total modifier content of 25 phr - significantly improves both strength and plasticity under all considered loading conditions. Specifically, tensile strength increased by 12.3%, bending strength by 22.2%, and compressive strength by 16.9%, while bending strain increased by 62.6%, tensile strain by 16.9%, and compressive strain by 1.7%.

To correlate the observed changes in mechanical properties with structural mobility, the glass transition temperatures (T_g) of selected compositions were measured by DSC [23]. The results are listed in Tab. 3.

The addition of DBT as well as BO_M to neat epoxy resin reduces the glass transition temperature (T_g) to the same extent (by 20 and 18 °C, respectively), which is typical for the use of plasticizers. At the same time, the strength decreases and the plasticity of the resulting epoxy polymer increases. However, the mechanisms of their interaction with the epoxy resin network are different: DBT, being an inert diluent, does not undergo chemical interaction, whereas BO_M chemically bonds to the epoxy matrix [23] and introduces flexible oligomeric segments, which also increase chain mobility. Thus, considering that both DBT and BO_M lead to comparable changes in thermal behavior (T_g) and mechanical performance, BO_M can serve as a reactive substitute for DBT.

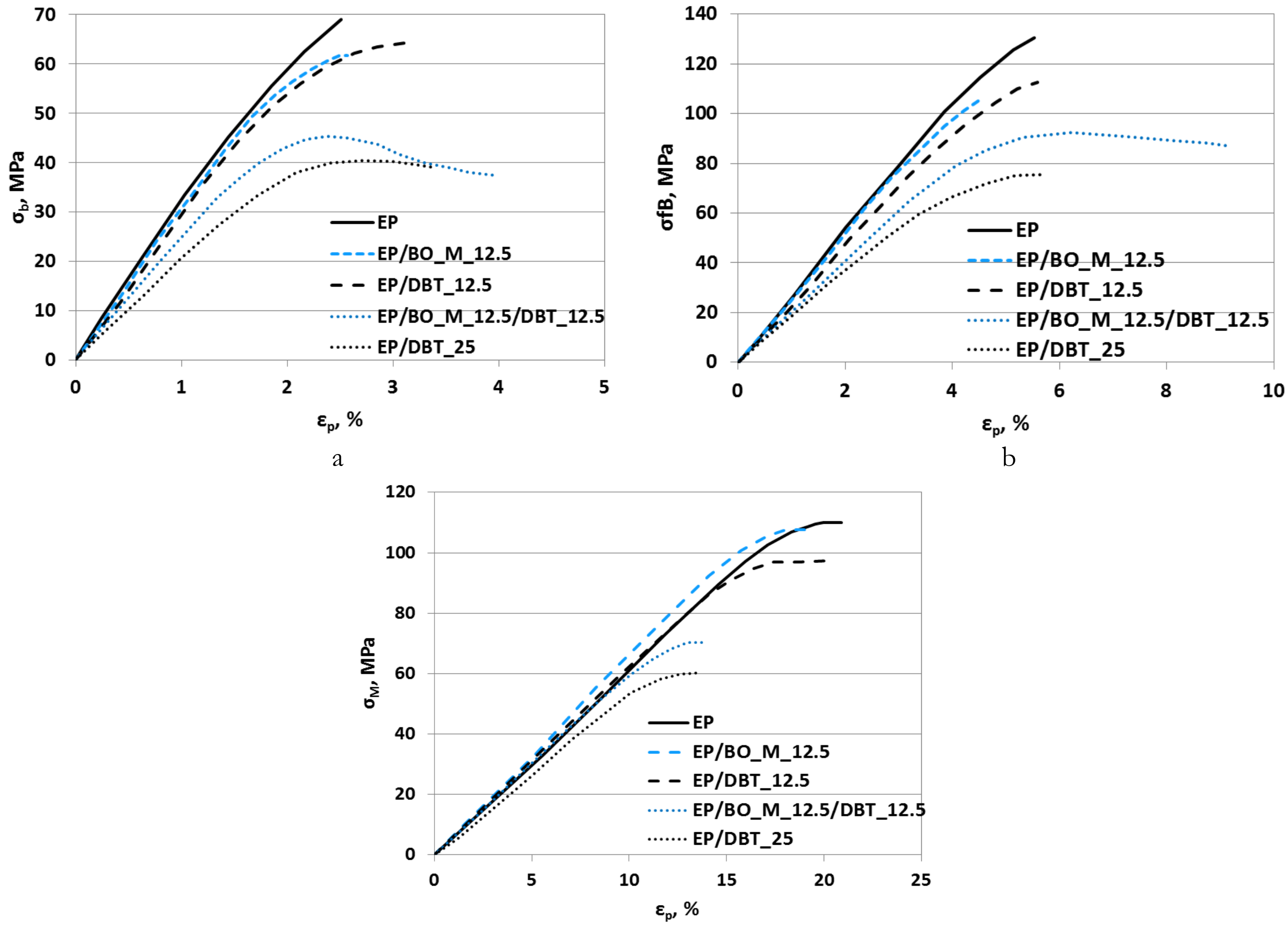

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Figure 4: Stress-strain behavior of neat and modified epoxy systems during (a) tensile, (b) three-point bending, and (c) compression testing

	T_g, °C
EP	112.3
EP/DBT_12.5	92.1
EP/BO_M_12.5	94.7
EP/BO_M_12.5/DBT_12.5	73.3

Table 3: Glass transition temperatures of selected epoxy formulations

The combined addition of EP/BO_M_12.5/DBT_12.5 leads to a substantial decrease in T_g (by 39 °C relative to the neat resin) while providing the highest deformation capacity. It should be noted that this decrease is due to the increase in total modifier content to 25 phr. The reduction of T_g is nearly additive (20 °C from DBT + 18 °C from BO_M = 38 °C), which probably indicates that the two plasticizing mechanisms operate without mutual interference. This maximal reduction in T_g is directly reflected in the highest deformation capacity (tensile strain 21.5%), as lower T_g facilitates segmental motion under load.

Therefore, BO_M represents a promising eco-friendly alternative to DBT as a standalone modifier. Furthermore, the results demonstrate the effectiveness of using a combination of BO_M and DBT to enhance the mechanical properties of epoxy systems. However, both plasticizers reduce the thermal resistance of the epoxy resin. Thus, the desired balance between thermal resistance and mechanical characteristics should be taken into account. Given that pyrolysis bio-oil is an underutilized by-product of pyrolysis, its use as a replacement for costly DBT can be considered economically viable.

Concentration-dependent strength properties of BO_M-modified epoxy

The investigation into the effect of BO_M modifier concentration on the strength of the epoxy composition under tension, compression, and three-point bending, presented in Fig. 5, demonstrate that the ultimate stress level increases with rising BO_M content up to 12.5 phr for all loading conditions considered. However, increasing the BO_M concentration further to 25 phr leads to a reduction in strength. Consequently, the optimal strength performance across all loading types is achieved with 12.5 phr of BO_M.

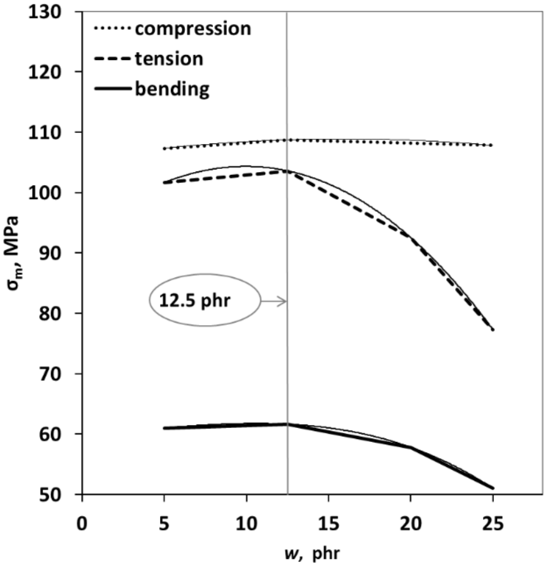

Figure 5: BO_M content-strength correlation in modified epoxy formulations.

It should be noted, that for compositions with BO_M content above 20 phr, a noticeable increase in viscosity and accelerated curing were visually observed, indicating an influence on the curing kinetics, which probably contributes to the strength reduction. Moreover, at such high concentrations, the oligomeric chains of the bio-oil may hinder the formation of a dense epoxy network. The curing kinetics of epoxy resins modified with pyrolysis bio-oil require further investigation. The approximation equations describing the correlation between the ultimate stress σ_m and the mass fraction w of the BO_M modifier for each loading type is presented in Tab. 4.

Loading type	Equation
Tension	$\sigma_m = -0.0014w^3 + 0.0129w^2 + 0.1982w + 59.891$
Three-point bending	$\sigma_m = -0.0131w^2 + 0.4177w + 105.58$
Compression	$\sigma_m = -0.0006w^3 - 0.0948w^2 + 2.0503w + 93.867$

Table 4: Correlation functions between ultimate strength and. bio-oil content

These equations enable the prediction of ultimate stress for any BO_M content within the studied concentration range.

CONCLUSIONS

The study demonstrates the viability of using pyrolysis bio-oils from plant waste as sustainable modifiers for epoxy resins.

Bio-oils from birch, sunflower, and mixed wood feedstocks can effectively modify epoxy resins. The resulting epoxy systems exhibit strength characteristics comparable to those modified with DBT, specifically achieving 94-97% of tensile strength, 73-97% of bending strength, and 11-39% higher compressive strength depending on the bio-oil type. The mixed wood-derived bio-oil (BO_M) provides the most balanced mechanical characteristics to the resulting polymer systems.

Simultaneous modification with BO_M and DBT (1:1 ratio) produced superior mechanical performance relative to the DBT-only reference (25 phr), with strength enhancements of 12% (tension), 22% (bending), and 16% (compression), accompanied by deformation increases of 17%, 62.5%, and 2%, respectively.

An optimal BO_M loading of 12.5 phr was determined, providing maximum strength under tension, bending and compression.

Thermal analysis revealed that both plasticizers reduce the thermal resistance of the modified epoxy systems. Consequently, the desired balance between thermal resistance and mechanical properties should be taken into account.

Thus, BO_M represents a promising eco-friendly alternative to DBT as a standalone modifier and in combination, providing significant enhancement of the mechanical properties of epoxy systems while offering economic benefits.

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