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Abstract

This study aimed at developing a lignin-based bio-binder from organosolv lignin extracted from mixed wood sawdust composed of three local wood species, namely Cordia, Iroko, and Passi, collected in Ngaoundéré, Cameroon, for application in particleboard manufacturing. The mixed sawdust was first sampled and characterized. Lignin was then extracted using the organosolv process, characterized, and modified by phenolation. The bio-binder formulation was optimized using response surface methodology based on a central composite design with three factors (lignin, furfural, and glycerol) and four responses (pH, viscosity, gel time, and insoluble fraction). The optimized bio-binder was subsequently used in the manufacturing process of particleboards produced from the mixed sawdust. The results showed that the mixed sawdust contained 45.6% lignin, with an extraction yield of 55.75% and a lignin purity of 91.1%. The optimized bio-binder exhibited a pH of 5.1, a viscosity of 342 mPa·s, a gel time of 3.35 min, and an insoluble fraction of 79.56%. The optimized particleboard exhibited a thickness swelling of 2.94%, an impact resistance of 100%, and a water absorption of 254%. Finally, the developed bio-binder contains 87.6% wood sawdust. When incorporated at 8.00% into particleboards containing 90.48% sawdust, it results in a final material with a wood sawdust content of 97.28%. These findings highlight the economic and industrial potentials of mixed wood sawdust as an abundant and readily available resource for the development of lignin-based bio-binders and more sustainable particleboards in Cameroon to promot local technology and economic development.

Key words : lignine, mixed wood sawdust, furfural, bio-binder, compacted particleboards.

1. Introduction

The global wood-based panel market is substantial and continues to expand, reaching USD 217.9 billion in 2024 and projected to reach USD 349 billion by 2034, with a compound annual growth rate (CAGR) of 4.8% (Global Market Insights Inc., 2025). This sustained growth is accompanied by a strong dependence on conventional synthetic adhesives, which are predominantly derived from petrochemical resources. Among these, urea–formaldehyde (UF) resins remain the most widely used adhesive systems in the wood-based panel industry, being employed in more than 90% of wood-based panels produced worldwide (Calvez et al. 2024). Other formaldehyde-based adhesive systems, including melamine–formaldehyde (MF), melamine–urea–formaldehyde (MUF), and phenol–formaldehyde (PF) resins, are also widely used depending on the type of panel and its intended application (Gonçalves et al. 2021; Calvez et al. 2024). Consequently, a large proportion of wood-based panels used in furniture, partitions, flooring, and other interior applications relies on synthetic adhesive systems associated with fossil-resource dependence and formaldehyde emissions. This issue is of particular concern because formaldehyde is a volatile compound with well-established adverse health effects. The International Agency for Research on Cancer (IARC) classified formaldehyde as carcinogenic to humans (Group 1) in 2006, based on sufficient evidence of an increased risk of nasopharyngeal cancer (IARC, 2006). Subsequent epidemiological evidence raised further concerns regarding a possible association between formaldehyde exposure and myeloid leukemia, although the evidence was considered insufficient to establish a causal relationship at that time (IARC, 2006). In response to the health risks associated with indoor formaldehyde exposure, the World Health Organization (WHO) established a guideline value of 0.1 mg/m³ for formaldehyde, averaged over 30 min, in its indoor air quality guidelines (WHO Regional Office for Europe, 2010). These environmental, health, and resource-related concerns have therefore intensified the search for renewable and technically viable alternatives to conventional petrochemical-based wood adhesives, particularly those derived from abundant lignocellulosic biomass (Gonçalves et al. 2021; Ait Benhamou et al. 2025).

Lignin represents a promising alternative to conventional formaldehyde-based adhesives for particleboard manufacturing. It is a major structural component of wood and is also present in lignocellulosic residues such as wood sawdust, which remains an underutilized industrial waste in Cameroon. The first- and second-stage wood-processing industries in Cameroon generate approximately one million cubic meters of sawdust annually (Tchuidjang et al. 2021). In

Ngaoundéré, this sawdust is mainly generated from Ayous, Sapelli, and Iroko wood species (Hassana et al. 2019). A significant proportion of this residue is burned, contributing to atmospheric pollution, while other quantities are used as fuel or applied in agricultural practices (Tchuidjang et al. 2021). Sawdust can also be incorporated into composting processes for agricultural applications. However, its slow biodegradability and high carbon-to-nitrogen ratio may limit its direct use as a soil amendment (Yaser et al. 2007). Beyond these conventional uses, lignocellulosic residues and, in particular, their lignin fraction can be chemically valorized into a wide range of value-added products, including fuels, chemicals, carbon-rich materials, dispersants, adhesives, and other functional materials (Bajwa et al. 2019). Nevertheless, the chemical valorization of wood residues remains relatively limited compared with their availability, despite their considerable potential for the development of value-added biomaterials. In the African context, and particularly in Cameroon, the main limitation is therefore not necessarily the chemical feasibility of such valorization, but rather its economic accessibility. The production of bio-based adhesives from wood residues may require activators, crosslinking agents, and functional additives that are costly to import but may be produced locally. Consequently, the value chain associated with Cameroonian sawdust remains largely underdeveloped, despite the abundant availability of this raw material and its considerable scientific and industrial potentials. This situation highlights the relevance of developing locally sourced bio-based adhesives as a strategy for increasing the value of wood-processing residues in Cameroon.

Chemical valorization of wood sawdust into bio-based binders can particularly benefit from its relatively high lignin content. Among technical lignins, organosolv lignin is considered an attractive feedstock for adhesive applications because it can be recovered with high purity, low ash and carbohydrate content, and without sulfur-containing groups, while retaining a relatively high content of chemically accessible functional groups (Kazzaz & Fatehi, 2020). These characteristics, together with the structural features of organosolv lignin, make it particularly suitable for further chemical derivatization to intensify some functional groups and the development of bio-based adhesive systems. Indeed, organosolv lignins obtained from different lignocellulosic feedstocks have been successfully investigated as wood adhesives, including in particleboard manufacture (Dababi et al. 2016; Koumba-Yoya et al. 2017). Recent research has further demonstrated the potential of combining lignin with furan-based compounds to reduce the reliance on petroleum-derived phenol and formaldehyde chemical substances. For instance, Tao et al. (2024) reported that lignin and furfural could replace up to 80% of phenol and 15%

of formaldehyde, respectively, in the synthesis of phenolic-type bio-based adhesives, while maintaining satisfactory bonding performance. Lignin-based adhesive systems have also been developed from lignins of different botanical and industrial origins, demonstrating that lignin source and structural characteristics can substantially influence adhesive reactivity and final performance (Sarazin et al. 2020 ; Younesi-Kordkheili & Pizzi, 2021; Singh et al. 2022). Furthermore, studies have demonstrated the feasibility of incorporating substantial proportions of lignin into wood adhesives and producing particleboards with satisfactory mechanical and physical properties (Mansouri et al. 2011; Koumba-Yoya et al. 2017). Furthermore, lignins derived from mixed hardwood feedstocks have also been characterized, demonstrating that heterogeneous wood sources can yield technically usable lignin fractions (Mun et al. 2021). However, these studies have generally focused either on the characterization of lignin obtained from mixed hardwoods or on the incorporation of pre-existing technical lignins into adhesive formulations. The direct valorization of a heterogeneous mixture of tropical wood sawdust through an integrated sequence involving organosolv lignin extraction, chemical modification, bio-binder formulation, and subsequent particleboard manufacture remains poorly documented. This issue is particularly relevant in Cameroon, where sawmill residues may originate from several tropical wood species processed simultaneously, making systematic separation of individual species difficult and limiting opportunities for species-specific valorization. Therefore, the present study investigates the development and optimization of a bio-based binder produced from organosolv lignin extracted from a heterogeneous mixture of tropical wood sawdust and its application in the manufacture of compacted particleboards for building and construction purposes. Beyond assessing the feasibility of this approach, the study aimed at establishing an integrated valorization pathway in which both the lignin fraction and the residual sawdust can be converted into a high-value wood-based material.

2. Material et methods

2.1. Sampling of wood sawdust

Wood sawdust samples were collected from sawmills in Ngaoundéré, Cameroon. The sawdust consisted of a mixture of three tropical wood species, namely Cordia (*Cordia platythyrsa* Baker), Iroko (*Milicia excelsa*), and Passi. The sawdust from the three species was not separated prior to sampling or subsequent processing. This approach was deliberately adopted to preserve the heterogeneous nature of the raw material and to avoid an additional separation step that

could increase processing costs and complicates the practical feasibility of the targeted valorization route at an industrial scale.

Sampling was carried out in three sawmills in Ngaoundéré on the same day that the wood was being pre-processed, thereby minimizing exposure of the sawdust to climatic conditions and preventing its mixing with material from previous processing batches. The three wood species were being processed simultaneously in the selected sawmills. Cordia (*Cordia platythyrsa* Baker) and Iroko (*Milicia excelsa*) were identified botanically, whereas Passi corresponds to a local vernacular name for which botanical identification has not yet been confirmed. These three species are locally available and are frequently processed in the Adamawa Region of Cameroon.

Several samples were collected from each of the three sawmills at different positions within the sawdust piles, including the bottom, middle, and top layers, in order to account for potential heterogeneity within the piles. The collected samples were manually homogenized to obtain a representative composite sample. The composite sample was then appropriately packaged and immediately transported to the Chemical Engineering Research Laboratory of the University of Ngaoundéré, Cameroon, for subsequent processing and characterization.



Figure 1 : Image of mixture sawdust of three tropical wood species

2.2. Wood sawdust pretreatment

The collected sawdust was initially sun-dried until a constant mass was reached. The dried sawdust was then mechanically ground and sieved through a 2 mm mesh. The material retained on the sieve was reground and sieved again under the same conditions. The fraction passing through the 2 mm sieve was collected to obtain a relatively homogeneous particle size (Ø < 2 mm) and stored in a moisture-protected environment until further use.

2.3. Physicochemical characterization of the sawdust

2.3.1. Determination of moisture content

The residual moisture content of the sawdust was determined in the laboratory according to the NF EN ISO 18134-1 standard (2016). The moisture content was used to correct all sample masses involved in yield calculations and to express the results on a dry-matter basis. Briefly, a known mass of the sample was dried at 105 °C in an isothermal oven until a constant mass was reached (approximately 24 h). After cooling in a desiccator, the samples were weighed again. The moisture content was calculated from the mass loss occurring during oven drying. The moisture content (MC), expressed as a percentage of the initial wet mass, was determined using Equation (1).

$$MC\ (\%) = \frac{m_1 - m_2}{m_1 - m_0} \times 100 \quad \ldots\ldots\ldots\ldots\ldots\ldots\ (1)$$

With m_0 being the mass of the empty capsule, m_1 capsule mass + wet sample and m_2 the mass after drying in the oven. The dry matter (DM) content is obtained by

$$DM = 100 - MC \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ (2)$$

2.3.2. Determination of ash content

The ash content (AC) of the sawdust was determined according to NF EN 14775 (2010). Briefly, a known mass of previously oven-dried sawdust (105 °C) was placed in a muffle furnace and heated at 550 °C. After incineration, the resulting ash residue was cooled in a desiccator and weighed. The ash content was calculated using Equation (3).

$$AC\ (\%) = \frac{m_3 - m_0}{m_1} \times 100 \quad \ldots\ldots\ldots\ldots\ldots\ldots\ (3)$$

where m_3 is the mass of the crucible after incineration and m_1 the mass of the initial dry sample.

2.3.3. Determination of Extractable Content

The extractable content was determined using a procedure adapted from ASTM D1107-21. Briefly, 3 g of oven-dried sawdust (m_2) were extracted with 150 mL of a toluene–ethanol mixture (2:1, v/v). After extraction, the sawdust residue was recovered by Büchner filtration, dried at 115 °C in an oven, and weighed (m_1) The extractive content (Ex), expressed as a percentage, was calculated from the mass loss of the sample during extraction according to Equation (4):

$$Ex\ (\%) = \frac{m_2 - m_1}{m_2} \times 100 \quad \ldots\ldots\ldots\ldots\ldots\ldots\ (4)$$

Where m_2 is the initial dry mass of the sawdust and m_1 is the dry mass of the sawdust residue after extraction.

2.3.4. Determination of acid-insoluble lignin content

The acid-insoluble lignin content of the sawdust was determined using the Klason method. Briefly, 1 g of oven-dried sawdust (m_5) was placed in a 1000 mL flask, and 30 mL of 72% H_2SO_4 was added. The mixture was continuously stirred at room temperature for 24 h. Subsequently, 560 mL of distilled water was added, and the resulting suspension was heated to boiling point under reflux for 2 h. After hydrolysis, the mixture was cooled and vacuum-filtered through a previously dried and pre-weighed Whatman glass-fiber filter. The insoluble residue retained on the filter was thoroughly washed with deionized water to remove residual acid and soluble compounds. The filtered material containing the insoluble residue was then dried at 105 °C to constant mass and weighed (m_6). The mass of acid-insoluble residue was obtained by subtracting the mass of the empty filter (m_f) from m_6. The acid-insoluble lignin (Klason lignin) content was calculated according to Equation (5).

$$L\,(\%) = \frac{m6 - mf}{m_5} \times (100) \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ (5)$$

2.3.5. Determination of cellulose content

The cellulose content was determined according to the method reported by Absadjieu et al. (2020). The method is based on the delignification of sawdust using ethanol and nitric acid. Briefly, 1 g of sawdust was mixed with 20 mL of 95% ethanol and 5 mL of concentrated nitric acid. The mixture was heated in a thermostatically controlled water bath at 95 °C for 1 h. After heating, the mixture was decanted and filtered. The treatment was then repeated under the same conditions. The operation was performed three times on the same sample. After the third treatment, the resulting white pulp was successively washed with a mixture of ethanol (40 mL) and nitric acid (10 mL), followed by 1 L of hot water. The resulting residue was then dried in an oven. The dried residue was considered to represent the cellulose fraction, and its content was calculated using Equation (6).

$$Cellulose\ content(C\%) = \frac{mass\ of\ cellulose}{mass\ of\ sawdust} \times 100 = \frac{m_f}{m_i} \times 100 \quad \ldots\ldots\ldots\ldots\ldots\ldots\ (6)$$

C= cellulose content (%); m_i = mass of dried sawdust ; m_f = mass of dried residue

2.3.6. Determination of hemicellulose content

The hemicellulose content was determined using the method developed by N'Diaye et al. (1996) and subsequently applied by Tchuidjang et al. (2021). Briefly, 1 g of sawdust (m_4) was mixed

with 150 mL of a 0.5M NaOH solution. Mixture was transferred to a flask fitted with a reflux condenser and heated under reflux at boiling temperature for 3 h 30 min. The resulting suspension was filtered through a Büchner funnel. The solid residue was washed and filtered four times with 150 mL of distilled water to remove Na^+ ions introduced by the NaOH solution. The washed residue was then dried at 105 °C and weighed (m_3). The hemicellulose content (H), expressed as a percentage, was determined from the mass loss of the sample following the alkaline treatment according to Equation (7).

$$H(\%) = \frac{m_4 - m_3}{m_4} \times \frac{100}{1} \quad \text{..... (7)}$$

2.4. Organosolv lignin extraction

Lignin was extracted from the sawdust using the organosolv process under the optimized conditions for reaction time, ethanol concentration, and temperature previously determined by Tchuidjang et al. (2021). Briefly, 140 g of oven-dried sawdust was mixed with 1064 mL of distilled water, 336 mL of 95% ethanol, 0.38 mL of concentrated H_2SO_4 (98%), and 0.38 mL of HCl (35%). After homogenization for 5 min, the mixture was transferred to a sealed 2-L stainless-steel extraction reactor, with approximately 1.4 L of reaction mixture loaded into the reactor. The reactor was subsequently placed in a MEMMERT oven and heated at 210 °C for 48 min.

At the end of the treatment, the reactor was removed from the oven and allowed to cool to room temperature before being opened in order to minimize solvent loss due to flash evaporation. The resulting extract was then sieved through a 100 µm mesh. The recovered fraction (filtrate) was diluted with 7 L of distilled water to induce lignin precipitation through an antisolvent effect. After 40 min of settling, the precipitated lignin, appearing as a dark-brown to brownish-black powder, was recovered by filtration through Whatman No. 4 filter paper. The recovered lignin was subsequently washed three times with 500 mL of distilled water adjusted to pH 6–7 and dried at 60 °C for 24 h. The lignin extraction yield (RR), expressed as a percentage, was calculated using Equation (8).

$$\boldsymbol{RL(\%) = \frac{mass\ of\ recovered\ lignin}{mass\ of\ sawdust \times L_Klason} \times 100 \quad \text{..... (8)}}$$

The purity of the extracted lignin was assessed using three complementary tests. These included a flame test, characterized by the formation of sooty black smoke and a characteristic phenolic odor; a water-insolubility test, combined with measurement of the pH of the resulting

suspension; and a solubility test in 1 mol/L NaOH, followed by gravimetric determination of the insoluble residue. The latter test was used as a semi-quantitative estimate of lignin purity calculated through equation (9).

$$P\ (\%) = \left(1 - \frac{mass\ of\ insoluble\ residu}{mass\ of\ Lklason}\right) \text{x } 100 \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (9)$$

2.4.1. Fractionation of lignin according to molecular weight

The crude organosolv lignin is a heterogeneous mixture of fractions with different molecular weights. The isolation of the most reactive fraction intended for phenolation (derivatization) and subsequent bio-binder formulation was carried out according to the method reported by Tindall et al. (2020), which exploits differences in solubility between lignin fractions with contrasting molecular weights. Briefly, 10 g of dry lignin was dissolved in a mixture of 95 mL of 96% ethanol and 5 mL of distilled water at 60 °C for 2 h. The resulting suspension was filtered, and the retained cake, corresponding to the heavy lignin fraction (Mn = 8,400 Da), was dried at 60 °C for 24 h. This fraction was subsequently used as the raw material for phenolation. The extracted phenol was directly used as the phenolating agent for this same lignin fraction.

The filtrate, corresponding to the light lignin fraction (Mn = 1,500 Da), was mixed with toluene at a 1:1 (v/v) ratio and subjected to liquid–liquid extraction. The organic phase was subsequently treated with a 10% NaOH solution (pH 12), homogenized, and subjected to a second phase separation. The resulting organic phase was then acidified dropwise with concentrated HCl, inducing precipitation of the bio-based phenol. The precipitated phenol was isolated by a final liquid–liquid extraction. These operations resulted in a fractionation yield of 95.4% and a bio-based phenol content of 1.2%.

2.4.2. Synthesis of bio-based phenolic compounds by lignin depolymerization

Lignin depolymerization to produce bio-based phenolic compounds was performed using zinc chloride ($ZnCl_2$) catalysis in ethanol, following the method reported by Zhang et al. (2014). Briefly, 20 g of dry lignin was mixed with 2.72 g of $ZnCl_2$ (0.02 mol) and 800 mL of 95% ethanol. The mixture was heated at 300 °C for 2 h. Under these conditions, the lignin macromolecular structure undergoes cleavage of interunit bonds, resulting in the formation of low-molecular-weight phenolic compounds. After cooling, the reaction mixture was filtered, and the filtrate was distilled at 78 °C to remove ethanol. The remaining material was subsequently evaporated to remove residual water. The resulting bio-based phenolic fraction was characterized in terms of yield, viscosity, and purity.

2.4.3. Chemical derivatization of lignin by phenolation

Phenolation was performed to increase the density of free phenolic hydroxyl groups in lignin through electrophilic aromatic substitution. The phenolation of lignin was carried out according to the method described by Pizzi et al. (1994). This modification step was intended to enhance the cross-linking reactivity of lignin with furfural during subsequent bio-binder formulation. Briefly, 20 g of dry lignin was first dissolved in 300 mL of 1 mol/L NaOH solution in a thermostatically controlled water bath at 60 °C for 1 h. The resulting solution was then homogenized with 200 g of bio-based phenol and 40 mL of 98% H_2SO_4, used as a catalyst. The mixture was subsequently heated at 120 °C for 2 h.

The hot reaction mixture was then poured into 5 L of ice-cold water. The resulting suspension was allowed to settle for 30 min and was subsequently filtered. The recovered cake was washed with hot water (65 °C; 3 × 500 mL) to remove unreacted phenol, followed by washing with 250 mL of 95% ethanol to remove residual organic compounds. The modified lignin was finally dried at 50 °C for 24 h. The resulting phenolated lignin was characterized by determining its free phenolic hydroxyl group content.

2.4.4. Determination of total phenolic content

The total phenolic content of the lignin samples was determined using the Folin–Ciocalteu colorimetric method. A calibration curve was prepared using gallic acid as the reference standard over a concentration range corresponding to 0–10 mg of gallic acid. Briefly, 0.2 mL of the lignin sample solution was mixed with distilled water and 0.2 mL of Folin–Ciocalteu reagent. After standing, 0.4 mL of 20% Na_2CO_3 solution was added, and the mixture was incubated in a water bath at 40 °C for 20 min. The absorbance was measured at 725 nm. The calibration curve was established from the linear relationship between absorbance and gallic acid concentration. The total phenolic content was expressed as milligrams of gallic acid equivalents per 100 g of dry matter (mg GAE/100 g DM) and calculated from the corresponding regression equation, taking into account the sample mass and residual moisture content.

2.5. Furfural synthesis from hemicellulosic residues

Furfural was produced from the hemicellulosic residues generated during the organosolv lignin extraction through a biphasic conversion process based on the modified method reported by Sweygers et al., (2018). This approach relies on the in situ extraction of furfural into an

immiscible organic solvent as it is formed, thereby limiting its degradation through resinification and improving the overall furfural yield.

Briefly, 50 g of hemicellulosic residues obtained from the organosolv lignin extraction were mixed with 1 L of distilled water and 40 mL of concentrated H_2SO_4 (98%), in the presence of 1 L of 1-pentanol as the extracting solvent. The resulting biphasic mixture was heated at 170 °C for 3 h. During the reaction, pentoses released through the acid hydrolysis of hemicelluloses were dehydrated to form furfural, which was simultaneously extracted into the organic phase. After cooling, the two phases were separated. The organic fraction was subjected to distillation at 137 °C using a condenser to recover 1-pentanol and obtain the crude furfural product, hereafter referred to as crude furfural.

2.6. Bio-binder formulation and optimization

Once the phenolated lignin and bio-based furfural had been produced, the proportions of the different constituents required for bio-binder formulation had to be determined. In addition, the appropriate sawdust-to-bio-binder ratio had to be established to produce a compact particleboard meeting the targeted dimensional and mechanical requirements.

These two optimization steps were conducted separately using the design of experiments (DoE) methodology. First, the bio-binder formulation was optimized based on four physicochemical performance specifications that had to be met before the formulation could be selected for particleboard manufacturing. The specifications defined for the bio-binder are presented in Table 1. The four parameters specified in the performance requirements were used as the four response variables in the experimental design for optimizing the bio-binder formulation.

In a second step, the optimized bio-binder was incorporated into mixed tropical hardwood sawdust, and the sawdust-to-bio-binder ratio was optimized for particleboard manufacturing. This second experimental design was based on three response variables, namely thickness swelling, water absorption, and impact resistance, which were used to assess the dimensional and mechanical performance of the resulting particleboards.

Tableau 1 : specifications defined for the bio-binder

Parameter	Specification	Standard / Method
pH	5.0 - 6.0	ASTM E70-19

Viscosity (mPa·s)	200 - 800	ASTM D1084-16
Gel time (min)	2 - 5	ASTM D3056-14
Insoluble residue (%)	≥ 81	Zhao et al., (2018)

2.6.1. Selection of factors and experimental domain

The optimization of the bio-binder formulation was performed using a design of experiments (DoE) approach based on a central composite design (CCD). Preliminary bio-binder formulation trials were conducted, from which three experimental factors were selected. The first factor was the modified lignin content (X_1), which forms the phenolic backbone of the cross-linked network. The second factor was the furfural content (X_2), which acts as the bio-based cross-linking agent. The third factor was the glycerol content (X_3), which was used as a plasticizing and viscosity-regulating agent. The experimental ranges selected for each factor are presented in Table 2. Four response variables were selected to characterize the cross-linking efficiency of the bio-binder: pH (Y_{pH}), viscosity (Y_{Visco}), gel time (Y_{GT}), and insoluble residue content (Y_{IRC})**.**

Table 2 : Experimental ranges selected the factors

Factors	Code	-α	-1	0	+1	+α
Modified Lignin content (%)	A	53.18	60	70	80	86.82
Furfural content (%)	B	1.48	10	22.5	35	43.52
Glycerol content (%)	C	1.59	5	10	15	18.41

Table 3 presents the actual value experimentation matrix of the centered composite experimental design implemented in the laboratory.

Table 3. Experimental design matrix of the central composite design

Run	**Coded values**			**Actual values (%)**		
	X_1	X_2	X_3	X_1	X_2	X_3
1	−1	−1	−1	60.00	10.00	5.00
2	+1	−1	−1	80.00	10.00	5.00

3	−1	+1	−1	60.00	35.00	5.00
4	+1	+1	−1	80.00	35.00	5.00
5	−1	−1	+1	60.00	10.00	15.00
6	+1	−1	+1	80.00	10.00	15.00
7	−1	+1	+1	60.00	35.00	15.00
8	+1	+1	+1	80.00	35.00	15.00
9	−α	0	0	53.18	22.50	10.00
10	+α	0	0	86.82	22.50	10.00
11	0	−α	0	70.00	1.48	10.00
12	0	+α	0	70.00	43.52	10.00
13	0	0	−α	70.00	22.50	1.59
14	0	0	+α	70.00	22.50	18.41
15	0	0	0	70.00	22.50	10.00
16	0	0	0	70.00	22.50	10.00
17	0	0	0	70.00	22.50	10.00
18	0	0	0	70.00	22.50	10.00
19	0	0	0	70.00	22.50	10.00

X_1 : modified lignin content, X_2 : furfural content ; X_3 : Glycerol content

For each experimental run, the amount of lignin specified by the corresponding formulation in the experimental design (Table 3) was mixed with 35 mL of distilled water. The pH was adjusted to 10–11 using 1 M NaOH, and the resulting solution was heated to 70 °C before the amount of furfural specified for the corresponding experimental run (Table 3) was added. After homogenization, the mixture was cooled to room temperature (25 °C), and its pH was adjusted to 5 using 10% HCl. The amount of glycerol specified by the corresponding experimental run (Table 3) was then added. After homogenization, the mixture was heated to 80 °C to obtain the bio-binder. Its cross-linking performance was subsequently evaluated through its application in the hot-pressing of the particleboards.

2.6.2. Characterization of the bio-binder

The resulting bio-binder was characterized in terms of pH, dynamic viscosity, gel time, and insoluble residue content. The pH of the bio-binder was measured using a pH meter according to ASTM E70-19. The pH provides information on the residual acidity of the medium, which is an important factor influencing the cross-linking kinetics.

The dynamic viscosity was determined using a rotational viscometer (Brookfield-type) according to ASTM D1084-16. Viscosity provides information on the ability of the bio-binder to coat and penetrate the sawdust during mixing. The measurement was performed by immersing spindle No. 3 in a 10 g sample of bio-binder.

The gel time was determined using a procedure adapted from ASTM D3056-14. Briefly, a drop of bio-binder was placed on a hot plate maintained at 150 °C, and the time required for the drop to transition from the liquid to the solid state was recorded. Gel time was used as an indicator of the available hot-pressing time during the application of the bio-binder in particleboard manufacturing.

The insoluble residue content was determined using a procedure adapted from Umemura et al. (2013). Briefly, the cured bio-binder was immersed in water for X min. After recovery from the aqueous medium, the sample was dried and weighed. The mass difference between the initial cured sample and the dried residue was used to determine the insoluble residue content. This parameter was considered an indirect indicator of the degree of cross-linking achieved within the polymer network.

2.7. Application of the bio-binder to the manufacture of compacted particleboards

The bio-binder formulation that best met the performance specifications defined for optimal cross-linking efficiency was selected for application in the particleboard hot-pressing experiments. The dimensional and physical performance requirements established for the particleboards are presented in Table 4.

Table 4. Dimensional and physical performance requirements for the particleboards

Parameter	Target specification	Reference standard	Test method
24h thickness swelling	≤ 15% (dry use) ≤ 18% (humid use)	EN 317	24h water immersion; thickness measurement before and after immersion

24h water absorption	≤ 30% (indicative target)	Derived indicator (non-standardized)	Mass measurement before and after immersion
Impact resistance (resilience)	Maximum value sought	In-house test (relative comparison)	Impact test

2.7.1. Selection of factors and experimental domain

The optimization of particleboard compaction was performed using a design of experiments (DoE) approach based on a central composite design (CCD). Only two experimental factors were considered necessary for this experiment: the proportion of dried sawdust (X_1) and the proportion of bio-binder (X_2). The experimental ranges investigated for each factor are presented in Table 5, while the experimental design matrix is presented in Table 6. Three response variables were selected to characterize the compaction performance of the particleboards : thickness swelling (Y_{TS}), impact resistance (Y_{IR}), and water absorption (Y_{WA}).

Table 5 : experimental ranges investigated of the factors

Factors	Code	-α	-1	0	+1	+α
Dried sawdust (%)	X_1	82.34	84	88	92	93.66
Bio-binder (%)	X_2	6.34	8	12	16	17.66

Table 6. Experimental design matrix and of the particleboard compaction design

Run	**Coded values**		**Actual values (%)**	
	X_1	X_2	X_1	X_2
1	−1	−1	84.00	8.00
2	+1	−1	92.00	8.00
3	−1	+1	84.00	16.00
4	+1	+1	92.00	16.00
5	−α	0	82.34	12.00
6	+α	0	93.66	12.00
7	0	−α	88.00	6.34
8	0	+α	88.00	17.66
9	0	0	88.00	12.00
10	0	0	88.00	12.00
11	0	0	88.00	12.00

12	0	0	88.00	12.00
13	0	0	88.00	12.00

X_1 : Dried sawdust ; X_2 : Bio-binder

For the particleboard compaction experiments, the amount of sawdust specified for each experimental run in the design (Table 6) was mixed with the corresponding amount of bio-binder specified by the same experimental run. The mixture was heated to 100 °C before being placed into the mold and pressed at 5 MPa. After demolding, the resulting compacted block was dried at 60 °C until constant mass was reached.

2.7.2. Characterization of the compacted particleboards

The compacted particleboards obtained were characterized by determining thickness swelling (Y_{TS}), water absorption (Y_{WA}), and impact resistance (Y_{IR}).

Thickness swelling was measured in accordance with EN 317. Briefly, particleboard specimens of known initial thickness were immersed in distilled water. After 24 hours of immersion, the thickness of each specimen was measured, and the thickness swelling was calculated from the change in thickness before and after immersion. This parameter was used to assess the dimensional stability of the particleboards under moisture exposure.

Water absorption was determined using the same immersion procedure as that used for the thickness-swelling measurement, but based on the difference in specimen mass before and after immersion in distilled water. This parameter was used as an indicator of the residual porosity and water uptake capacity of the sawdust–bio-binder network.

Impact resistance was evaluated using a free-fall impact test based on NF EN 14323. Briefly, a mass was dropped from a height of 1 m onto a metal plate placed on the specimen. The impact energy transmitted to the specimen was controlled by maintaining a constant drop height.

2.7.3. Modeling of the bio-binder formulation and particleboard manufacturing processes

The model postulated for both experimental designs to relate each response variable (Y) to the corresponding experimental factors was a second-order polynomial model including interaction terms, as expressed by Equation (10):

$$Y = \beta_0 + \Sigma \beta_i x_i + \Sigma \beta_{ii} x_i^2 + \Sigma \beta_{ij} x_i x_j + \varepsilon \qquad (10)$$

where β_0 is the model intercept; β_i, β_{ii}, and β_{ij} are the linear, quadratic, and interaction coefficients, respectively; x_i_represents the coded variables; and ε is the residual error.

The models were subsequently reduced by removing statistically non-significant terms. The adequacy of each model was assessed using several statistical indicators, including the coefficient of determination (R^2), calculated using Equation (11), the absolute average deviation mean (AADM), calculated using Equation (12), the bias factor (Bf), calculated using Equation (13), and the accuracy factor (Af), calculated using Equation (14).

$$R^2 = \frac{\Sigma iN(\hat{Y}i,calc - \bar{Y})^2}{\Sigma iN(Yi,exp - \bar{Y})^2} \qquad (11)$$

$$AADM = [\, \Sigma_i^N(|Y_{i,exp} - Y_{i,calc}| / Y_{i,exp}) \,] / N \qquad (12)$$

$$B_f = 10^B \qquad (13) \qquad \text{with } B = (1/N)\, \Sigma i\, log(Yi,calc / Yi,exp)$$

$$A_f = 10^A \qquad (14) \qquad \text{with } A = (1/N)\, \Sigma i\, |log(Yi,calc / Yi,exp)|$$

With $Y_{i,exp}$ the experimental response, Yi,calc the response calculated by the model for trial i, $\bar{Y}$ the mean of the experimental responses and N the total number of trials.

The statistical significance of the effects was also assessed by Fisher's test (ANOVA), which compares the mean square of the regression to the experimental error.

3. Results

3.1. Characteristics of the heterogeneous sawdust mixture of tropical wood species and its extracted lignin

Table 7 and Figure 1 present the physicochemical characteristics of the heterogeneous sawdust mixture of tropical wood species (Cordia, Iroko, and Pasi) used in this study.

Table 7. Physicochemical characteristics of the heterogeneous sawdust mixture

Parameter	Content (%)
Moisture content	8.25 ± 0.11
Dry matter sèche	91.75 ± 0.27
Ash	2.76 ± 0.07
Cellulose	34.50 ± 0.50
Klason lignin	45.60 ± 0.04
Extractives	2.99 ± 0.00
Hemicelluloses*	16.91 ± 0.00

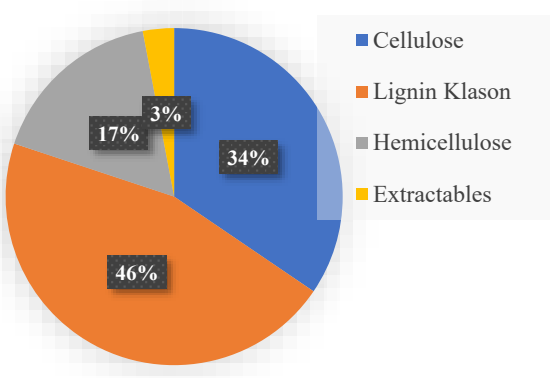


Figure 1: Chemical composition of sawdust from heterogeneous wood species (Cordia, Iroko, and Passi)

These results show that lignin, the main component sought for the production of the bio-binder, is predominant in the raw material (sawdust). Table 8 shows the characteristics of the lignin extracted from the heterogeneous sawdust mixture.

Table 8 : Characteristics of lignin extracted from Organosolv sawdust of heterogeneous wood species (Cordia, Iroko, and Pasi).

Parametre	Values obtained	Litterature	Source
Extraction yield (%)	55.75 %	35 - 65	Zakzeski et al., 2010
pH	4.1	3.5 - 5.0	Pan et al., 2006
Purety (%)	91.07	88 - 96	Dababi et al., 2016
Insolubility rate (%)	90.67	> 85	Pan et al., 2006

Table 8 shows a satisfactory lignin extraction rate compared to data in the literature. The extracted lignin has a purity of 91%. The 9% impurities correspond to cellulose microparticles co-precipitated during filtration and residual minerals included in the ash content (2.76%).

The pH of 4.1 in aqueous suspension confirms the naturally acidic nature of this lignin, linked to its partially dissociated carboxylic and phenolic groups. Figure 2 shows the extracted lignin and its fractionation composition by solubility as a function of molecular weight.

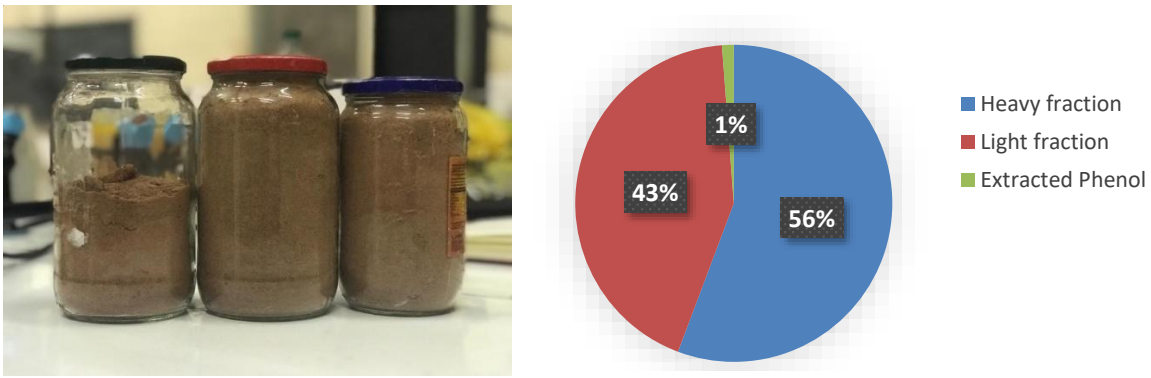


Figure 2: (a) Image of lignin extracted from sawdust of heterogeneous wood species (Cordia, Iroko and Pasi) and (b) the composition of its fractional separation as a function of molecular mass

Figure 2(b) shows that the heavy fraction is the predominant fraction. This is the fraction used as the main raw material for modifying lignin with phenol and for formulating the bio-binder.

The light fraction, richer in free phenols with low molecular weight, allowed for the extraction of 1% of bio-based phenol from the crude lignin. To increase the crosslinking potential of the crude lignin, it was modified with phenol, and the results are shown in Table 9.

Tableau 9. Variation of total phenolic content following lignin phenolation

Parameter	Experimental value (mg GAE/100 g DM)	Liu et al. (2024) (mmol/g)
Total phenolic content before phenolation	582.267	1.49
Total phenolic content after phenolation	807.733	1.93
Absolute increase	**+225.466**	+44
Relative increase (%)	**38.72**	**29.5**

DM: *dry matter;* ***GAE:*** *gallic acid equivalents*.

This result shows that the phenolation increased the phenolic hydroxyl content of the extracted lignin from 582.267 to 807.733 mg/100 g DM, corresponding to a relative increase of 38.72%. This increase is consistent with the enhancement of phenolic hydroxyl groups reported for phenolated hardwood kraft lignin, for which Liu et al. (2024) reported an increase from 1.49 to 1.93 mmol/g (29.5%). This makes this modified lignin a better-prepared raw material for the crosslinking step.

3.2. Development of the bio-based adhesive

The bio-based adhesive was developed using furfural produced from the hemicellulosic fraction of heterogeneous tropical wood sawdust composed of *Cordia*, *Milicia* (iroko), and Passi. The resulting furfural had a pH of 4.1 and was obtained with a yield of 80%. It was subsequently reacted with phenolated lignin to produce the bio-based adhesive according to a central composite experimental design.

3.2.1. Mathematical modeling of the responses characterizing bio-based adhesive performance

The experimental responses obtained from the central composite design used for bio-based adhesive development are presented in Table 10. The statistical indicators used to assess model adequacy are presented in Table 11.

Table 10. Experimental responses of the central composite design for bio-based adhesive development

Run	X_1	X_2	X_3	Y_{pH}	Y_{Visco}	Y_{GT}	Y_{IR}

	(%)	(%)	(%)		(mPa·s)	(min)	(%)
1	80	10	15	5.20	439.2	4.70	80.47
2	80	35	15	4.68	1217.6	8.53	78.92
3	70	22.5	18.41	5.19	660.4	6.75	78.90
4	60	10	15	5.85	271.0	2.65	79.99
5	80	35	5	5.71	551.2	5.48	75.28
6	60	35	15	5.26	729.8	6.68	75.25
7	70	22.5	10	5.66	370.9	4.55	79.26
8	70	22.5	10	5.83	421.9	4.53	78.47
9	86.82	22.5	10	5.30	635.7	6.98	77.73
10	70	22.5	10	5.27	311.3	4.48	77.16
11	70	22.5	10	5.73	460.7	3.97	77.35
12	70	22.5	1.59	6.67	351.8	5.13	77.69
13	80	10	5	5.74	201.8	3.67	80.71
14	60	10	5	6.90	58.7	3.10	80.79
15	70	22.5	10	5.73	406.8	5.38	80.11
16	60	35	5	6.46	123.6	4.87	73.50
17	70	43.52	10	5.14	675.0	5.53	77.23
18	53.18	22.5	10	6.40	333.9	5.05	75.21
19	70	1.478	10	6.21	232.5	1.23	82.88

X_1 = modified lignin; X_2 = furfural; X_3 = glycerol; Visco = dynamic viscosity; GT = gel time; IR = insoluble residue content.

Table 11. Statistical indicators for assessing the adequacy of the mathematical models

Indicators	pH	Viscosity	Gel-time	Insoluble residue	Acceptance criterion
R^2	0.95	0.91	0.97	0.92	≥ 0.90
Ajusted R^2	0.90	0.86	0.95	0.83	≥ 0.80
AADM	0.10	0.64	0.18	0.48	[0 – 0.3]
Af	1.02	1.25	1.05	1.01	[0.75 – 1.25]
Bf	1.00	1.03	0.99	1.00	[0.75 – 1.25]

Overall, Table 11 shows that the R^2 values > 0.90, together with the corresponding adjusted R^2 values > 0.80, indicate good agreement between the models and the different responses. Furthermore, the bias and accuracy factors, which were close to 1, indicate that the deviations between the predicted and experimental values were very narrow. Evaluation of model quality based on AADM showed good predictive performance for pH and gel time. In contrast, the AADM values > 0.30 obtained for viscosity and insoluble residue content indicate a substantial average deviation between the predicted and experimental values. The data from the analysis of variance (ANOVA) are presented in Table 12.

Tableau 12 : Analyses de la variance du modèle mathématique

Responses	F	p (model)	p (lack of adjustment)
pH	21.69	< 0.0001	0.781 (n.s.)
Viscosity	19.11	< 0.0006	0.092 (n.s.)
Gel-time	35.60	< 0.0001	0.914 (n.s.)
Insoluble residue	11.08	0.0007	0.915 (n.s.)

The results show that Fisher's F-values range from 11.08 to 35.60, associated with p-values ≤ 0.0007. These p-values < 0.05 confirm the overall significance of the model for predicting the four responses. This confirms that the quadratic polynomial accurately accounts for the experimental variability observed in the studied domain. The model is therefore statistically validated, and its responses can be optimized. The polynomial equations that illustrate the influence of the different factors on the responses are expressed using polynomials equations (15, 16, 17, and 18).

$$Y_{pH} = 5{,}65 - 0{,}37X_1 - 0{,}25X_2 - 0{,}46X_3 + 0{,}09\ X_1\ X_3 - 0{,}08\ X_2\ X_3 + 0{,}05\ X_1^2 - 0{,}01\ X_2^2 + 0{,}08\ X_3^2 \ldots\ldots\ldots\ldots\ldots\ldots (15)$$

$$Y_{Visco} = 444{,}93 + 126{,}99\ X_1 + 175{,}42\ X_2 + 164{,}12\ X_3 + 75{,}51\ X_1\ X_2 + 10{,}66\ X_1\ X_3 + 102{,}86\ X_2\ X_3 \ldots\ldots\ldots\ldots\ldots\ldots (16)$$

$$Y_{GT} = 4{,}59 + 0{,}61\ X_1 + 1{,}37\ X_2 + 0{,}60\ X_3 - 0{,}02\ X_1\ X_2 + 0{,}34\ X_1\ X_3 + 0{,}54\ X_2\ X_3 + 0{,}47\ X_1^2 - 0{,}46\ X_2^2 + 0{,}44\ X_3^2 \ldots . (17)$$

$$Y_{IR} = 78{,}48 + 0{,}74\ X_1 - 2{,}09\ X_2 + 0{,}47\ X_3 + 0{,}63\ X_1\ X_2 + 0{,}31\ X_1\ X_3 + 0{,}80\ X_2\ X_3 - 0{,}74\ X_1^2 + 0{,}53\ X_2^2 - 0{,}10\ X_3^2 \ldots\ldots (18)$$

These equations show, on the one hand, the influences of independent factors on the four responses and, on the other hand, the influences of interactions on the responses.

3.2.2. Influence of independent factors on the characteristics of the bio-binder produced

Figure 3 shows the effects of the main factors on the characteristics of the bio-binder.

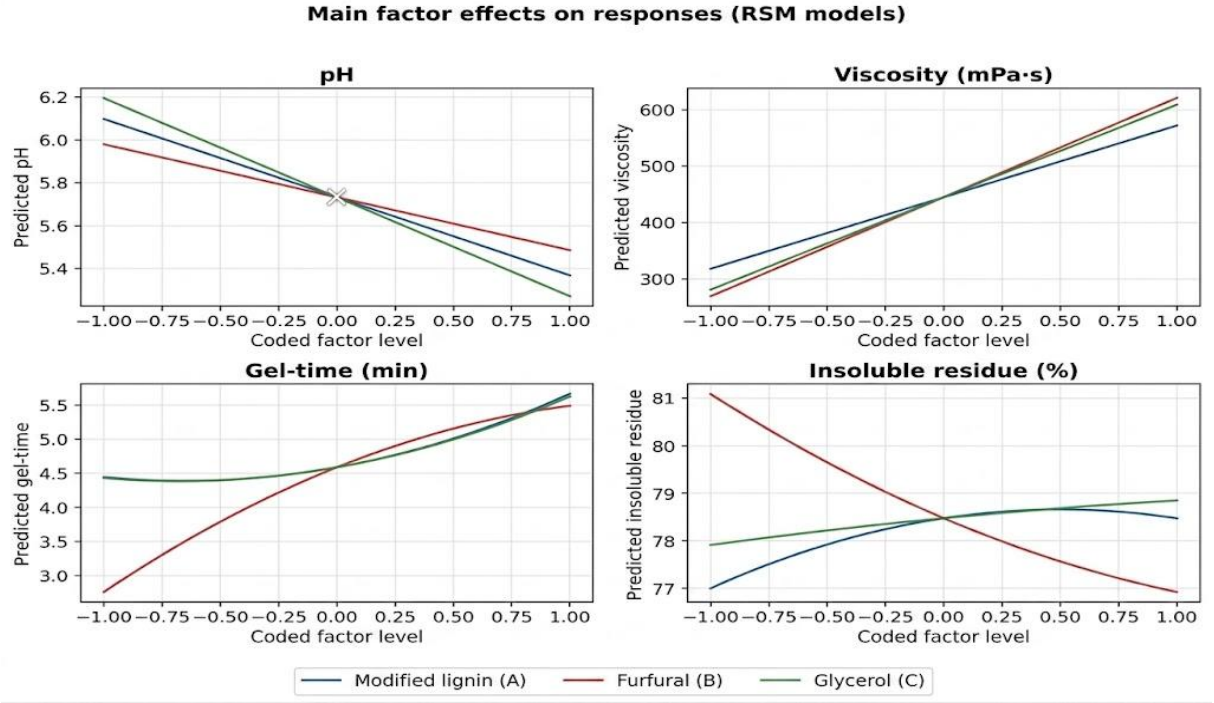


Figure 3: Effects of the main factors on the pH (a), viscosity (b), gel time (c) and insolubility rate (d) of the bio-binder

The results presented in Figure 3a show that increasing the contents of modified lignin (X_1), furfural (X_2), and glycerol (X_3) was generally associated with a decrease in the pH of the bio-binder. This trend indicates a shift of the medium toward more acidic conditions as the proportions of the different constituents increased within the experimental domain. In contrast, Figure 3b shows an overall increase in bio-binder viscosity with increasing levels of the three

factors. The effect of furfural appeared particularly pronounced, while modified lignin and glycerol also exhibited increasing trends, with some evidence of nonlinear variation within the experimental domain.

Regarding gel time (Figure 3c), the effects of the factors appeared more complex. Increasing the modified lignin content did not result in a monotonic change in gel time. Rather, gel time initially decreased and then increased at higher lignin contents. Furfural and glycerol also exhibited nonlinear behavior, with an increase in gel time over a large portion of the experimental domain. These observations suggest the presence of quadratic effects, which should be confirmed by analysis of the mathematical models and ANOVA results.

Figure 3d indicates a slight tendency toward an increase in the insoluble residue content with increasing modified lignin content and, to a lesser extent, glycerol content. Conversely, increasing the furfural content was generally associated with a decrease in insoluble residue content, followed by a slight increase at the highest furfural level. Thus, among the three factors investigated, furfural appeared to produce the most pronounced variations in several of the responses characterizing the bio-binder. However, this descriptive observation alone does not allow a statistically significant effect or the predominance of furfural to be established ; such conclusions should be based on the model coefficients and statistical analysis of their significance.

Overall, the results highlight differentiated and, in some cases, nonlinear effects of the factors on the properties of the bio-binder. They also reveal trade-offs among the different responses specified in the performance requirements, thereby justifying the use of a multi-response optimization approach to determine the optimum formulation conditions.

3.3. Development of the Compact Particleboard

3.3.1. Mathematical Models of the Responses Characterizing the Produced Particleboard

The responses obtained from the experimental design used for the production of the particleboard are presented in Table 13. The statistical indicators used to assess the validity of the mathematical models describing the particleboard compaction process with the formulated bio-binder are presented in Table 14.

Table 13. Experimental matrix of the particleboard compaction design using the formulated bio-binder

Run	X_1 (%)	X_2 (%)	Y_{TS} (%)	Y_{IC} (%)	Y_{WA} (%)
1	88	17.66	19.946	96.523	446.391
2	88	12	9.970	98.966	306.580
3	93.66	12	9.324	96.532	298.250
4	88	12	10.326	99.010	290.233
5	92	8	0	97.541	233.006
6	88	12	10.145	99.017	289.375
7	84	16	21.389	98.000	425.195
8	82.34	12	16.395	98.979	491.755
9	88	6.34	10.462	99.485	285.039
10	88	12	10.000	98.918	284.323
11	88	12	9.722	100.000	301.671
12	84	8	15.298	100.000	350.792
13	92	16	22.914	96.248	397.666

X_1 = wood sawdust; X_2 = bio-binder; TS = thickness swelling; IC = impact resistance; WA = water absorption.

Table 14. Statistical indicators for assessing the validity of the mathematical models describing particleboard compaction

Response	R^2	Ajusted R^2	AADM	Af	Bf
Thickness swelling	0.927	0.875	0.070	1.075	0.981
Impact resistance	0.950	0.915	0.002	1.002	1.000
Water absorption	0.944	0.903	0.038	1.038	1.001
Reference criteria	> 0.90	> 0.80	[0 – 0.3]	[0.75 – 1.25]	[0.75 – 1.25]

The results presented in Table 14 show that the R^2 and adjusted R^2 values, which were greater than 0.90 and 0.80, respectively, indicate good agreement between the model predictions and the three responses (YGonf, YRC, and YAE). The AADM values, all below 0.30, further indicate that the models can reliably predict the different responses. The Af and Bf values, which were close to 1, confirm the absence of substantial systematic bias across the experiments. The polynomial equations describing the effects of the different factors on the responses are given by Equations (19), (20), and (11), while the results of the analysis of variance (ANOVA) performed on the mathematical models are presented in Table 15.

$$Y_{TS}\,(\%) = 1151{,}50 - 21{,}83X_1 - 26{,}01X_2 + 0{,}26\,X_1\,X_2 + 0{,}10\,X_1^2 + 0{,}18\,X_2^2 \ldots (19)$$

$$Y_{IR}\,(\%) = -207{,}49 + 7{,}29\,X_1 - 0{,}35\,X_2 + 0{,}01\,X_1\,X_2 - 0{,}04\,X_1^2 - 0{,}03\,X_2^2 \ldots\ldots (20)$$

$$Y_{AW}\,(\%) = 23881{,}85 - 504{,}22\,X_1 - 152{,}20\,X_2 + 1{,}41\,X_1\,X_2 + 2{,}69\,X_1^2 + 1{,}78\,X_2^2 \ldots (21)$$

Tableau 15 : niveaux de significativité des facteurs du modèle (seuil α = 0,05)

Parameter	Thickness swelling	Impact resistance au Choc	Absorption water
Model	0.0007 **	0.0002 **	0.0003 **
X_1	0.0065 **	0.0002 **	0.0005 **
X_2	0.00025 **	0.0002 **	0.0002 **
$X_1\,X_2$	0.0065 **	0.3846 ns	0.1032 ns
X_1^2	0.1699 ns	0.0034 **	0.0034 **
X_2^2	0.0121 **	0.0055 **	0.0170 **
Manque d'ajustement	0.00007 **	0.8381 ns	0.0132 **

*X_1 = wood sawdust; X_2 = bio-binder; $p < 0{,}05$; ** = significant; ns = not significant*

The analysis of variance showed that the three mathematical models were highly significant, with *p*-values ranging from 0.0002 to 0.0007. The linear effects of sawdust content (X_1) and bio-binder content (X_2) were significant for all three responses studied ($p < 0.05$). The X_1X_2 interaction was significant for thickness swelling ($p = 0.0065$), but did not have a statistically significant effect on impact resistance ($p = 0.3846$) or water absorption ($p = 0.1032$). The quadratic terms further showed that X_1^2 contributed significantly to the variability of impact resistance and water absorption, whereas X_2^2 was significant for all three responses. These results confirm the presence of linear and quadratic effects and, in the case of thickness swelling, an interaction effect between the factors on the properties of the particleboards.

The lack-of-fit analysis, however, showed different behaviors depending on the response. The lack of fit was not significant for impact resistance ($p = 0.8381$), indicating a satisfactory agreement between the model and the experimental data for this response. In contrast, the lack of fit was significant for thickness swelling ($p = 0.00007$) and water absorption ($p = 0.0132$), indicating that the quadratic model adopted did not fully account for the experimental variability observed for these two responses. Despite this limitation, the high coefficients of determination ($R^2 = 0.927–0.950$) and the low AADM values (0.002–0.070) indicate an overall good descriptive performance of the models. The models can therefore be used to explore the

experimental domain and identify favorable formulation conditions, with particular caution regarding predictions of thickness swelling and water absorption**.**

3.3.2. Influence of independent factors on the characteristics of the compacted particle board

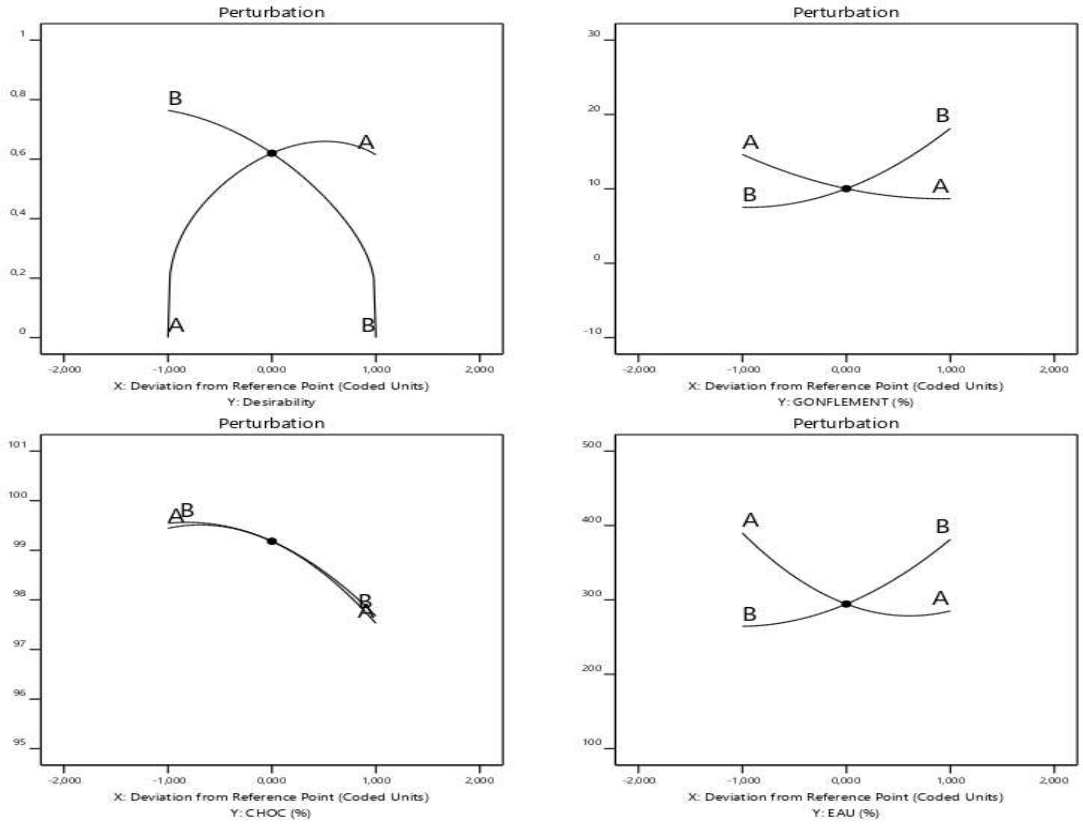


Figure 4: Different effects of factors around the central point of the plane on the swelling (a), impact resistance (b) and water absorption (c) of the compacted particle board.

This figure shows that sawdust is the main factor whose increased content in the formula positively influences the reduction of swelling (Figure 5a) in the compressed particle board and its water absorption capacity (Figure 5c). However, Figure 5b shows a negative influence of increased sawdust and binder on the impact resistance of the compressed particle board. Overall, Figure 5d shows that sawdust is the main factor whose increased content contributes positively to obtaining the desired compressed particle board.

4. Discussion

4.1. Physicochemical characteristics of the heterogeneous wood sawdust used and its extracted lignin

The results showed that, with a moisture content of 8%, the heterogeneous wood sawdust used in this study remained well below the 15% threshold generally considered suitable for storing lignocellulosic biomass without a significant risk of microbial fermentation or degradation (Mani et al. 2006). This value also falls within the range of 7–12% reported for air-dried tropical wood sawdust in the Sudano-Sahelian zone (Tchuidjang et al. 2021). The ash content obtained (2.76 ± 0.07%) was higher than the 1.20% reported by Tchuidjang et al. (2021) for *Ayous* sawdust from Ngaoundéré, but it remained within the 0.5–5% range reported for African tropical woods (Akpan & Adeosun, 2019). The increase of more than 1.5 percentage points may be partly attributed to *Iroko*, which is known to contain relatively high levels of silica and mineral matter (Ona et al., 1995). However, the contribution of *Passi* cannot be ruled out, as this species remains poorly documented in the available literature. For the subsequent stages of this study, the relatively low ash content (<5%) is considered favorable, since excessive mineral matter may interfere with acid catalysts and adversely affect lignin extraction during the organosolv process (Zhao et al. 2009).

The hemicellulose content of the heterogeneous wood sawdust (16.91%) was lower than that generally reported for African tropical woods (approximately 18%). This may be attributed to partial degradation during prolonged solar drying of the heterogeneous sawdust. Indeed, hemicelluloses constitute the most thermally sensitive fraction among the main chemical components of lignocellulosic biomass (Mani et al. 2006).

The cellulose content of the heterogeneous wood sawdust (34.50%) was lower than that reported for *Ayous* (45.33%) by Tchuidjang et al. (2021). However, it should be noted that the reported value falls below the 40–50% range generally associated with African tropical woods (Akpan & Adeosun, 2019; Betene et al. 2018). In contrast, the Klason lignin content (45.60%) determined for the heterogeneous wood sawdust was considerably higher than the values reported for *Ayous* (30.32%), *Sapelli* (28–32%), and *Iroko* (25–30%) (Akpan & Adeosun, 2019; Tchuidjang et al. 2021). This suggests that the use of a mixture of wood species may contribute to a relatively high lignin content, which is particularly advantageous in the present study because lignin constitutes the main raw material targeted for bio-binder production.

The extractable content (2.99%) was associated with the presence of volatile compounds, which was negligible in the present material (0.003%). This result indicates that the heterogeneous wood sawdust contained very low amounts of free waxes, resins, and tannins. This finding is therefore favorable for obtaining relatively pure lignin during the subsequent extraction process (Dababi et al. 2016).

Furthermore, the lignin extracted from the heterogeneous wood sawdust exhibited a purity of 91.07%, which can be considered satisfactory, as it falls within the reported range of 88–96% for ethanol-organosolv lignins obtained to date (Dababi et al. 2016; Pan et al. 2006). Organosolv lignin is generally regarded as a relatively pure technical lignin, mainly because it contains little or no sulfur, thereby preserving a greater proportion of free phenolic groups, whereas these groups may be chemically modified or partially blocked during kraft or sulfite pulping. The pH of 4.1 measured for the aqueous lignin suspension further confirms its naturally acidic character, which may be associated with carboxylic and partially dissociated phenolic groups. A pH slightly below the commonly reported range of 3.5–5 may result from residual traces of hydrochloric or sulfuric acid remaining after washing, or from a relatively higher density of free acidic groups in the lignin extracted from these Cameroonian wood species (Pan et al. 2006).

The light fraction, which was richer in low-molecular-weight phenolic compounds, enabled the recovery of 1.2% biosourced phenol through toluene extraction followed by basification and acidification. This yield is comparable to the 1.49% phenol yield reported by Zhang et al. (2014) during the catalytic depolymerization of lignin using $ZnCl_2$ in ethanol at 300 °C for 2 h. This close agreement suggests that the fractionation and subsequent recovery procedure used in the present study enabled the effective recovery of low-molecular-weight phenolic compounds from the light lignin fraction, although differences in lignin origin and processing conditions should be considered when comparing the two results.

4.2. Phenolated lignin

The extracted lignin was effectively modified by phenolation, resulting in a **38.72% increase in its phenolic hydroxyl content**. More specifically, under the acidic conditions provided by sulfuric acid, the phenol underwent acid-catalyzed activation, generating electrophilic species capable of reacting with activated aromatic positions of the lignin structure and thereby promoting the formation of new aromatic C–C bonds. The net increase of **225.466 mg/100 g dry matter** in phenolic hydroxyl groups (Table 10) reflects the enhanced phenolic functionality of the modified lignin. This increase provides a greater number of potentially reactive sites for subsequent condensation with furfural during bio-binder formulation. Consequently, the higher phenolic functionality of the modified lignin makes it a more suitable precursor for the subsequent crosslinking step with furfural.

The **38.72% increase in phenolic hydroxyl content** is consistent with the results reported by Liu et al. (2024) (Table 9), supporting the effectiveness of the phenolation treatment applied in the present study. The observed increase in phenolic hydroxyl groups indicates that the treatment successfully enhanced the phenolic functionality of the lignin, which is expected to favor its subsequent reactivity toward furfural during bio-binder formation.

4.3. Influence of factors on the properties of the bio-binder produced

4.3.1. Effect of lignin, furfural, and glycerol on pH

Figure 3a shows that increasing the levels of the three factors lignin, furfural, and glycerol promotes a decrease in the pH of the bio-binder. This behavior can be attributed to the ability of these three components to contribute to proton generation or to modify the acid-base equilibrium within the formulation. Lignin, for example, is an amorphous macromolecular polymer rich in oxygen-containing functional groups, particularly phenolic hydroxyl groups (-OH) and carboxylic groups (-COOH). Although these groups are weakly acidic, increasing the lignin content directly increases the concentration of potentially dissociable acidic sites in the medium. Under the formulation conditions, partial dissociation of these groups contributes to an increase in the concentration of hydronium ions (H_3O^+), thereby lowering the pH of the bio-binder.

A similar trend may be observed with furfural, a heterocyclic aldehyde produced by the dehydration of pentoses. Under the heating conditions used in the present formulation (80 °C), furfural may undergo oxidation and degradation reactions leading to furoic acid and other low-molecular-weight organic acids. Consequently, increasing the furfural proportion may increase the overall acidic character of the formulation, contributing to the observed decrease in pH.

Glycerol, a triol ($C_3H_8O_3$) and polar protic solvent, may also contribute indirectly to this behavior. Increasing its concentration modifies the physicochemical environment of the formulation, particularly its polarity and dielectric properties, which can affect the dissociation equilibrium of weak acidic groups originating from lignin and acidic impurities or degradation products present in furfural. This may increase the effective concentration of free protons in the medium and consequently decrease the pH.

Overall, the individual and combined effects of these three constituents appear to converge toward the formation of a formulation with a greater concentration of acidic and polar reactive species. The naturally acidic character of lignin, associated with its phenolic and carboxylic

groups, together with the intrinsic acidity and/or oxidation products of furfural, is therefore reinforced by the solvent and physicochemical effects of glycerol.

4.2.2. Effect of lignin, furfural, and glycerol on viscosity

Figure 3b also shows that increasing the levels of lignin, furfural, and glycerol, either individually or simultaneously, results in a significant increase in the viscosity of the bio-binder. This behavior can be attributed to changes in the molecular structure, intermolecular interactions, and the onset of polymerization reactions within the formulation. Lignin is a high-molecular-weight, highly branched polymer with a relatively rigid structure composed of phenylpropane units. As its concentration increases, the number of macromolecular chains per unit volume also increases. These large molecules generate substantial steric hindrance and become increasingly entangled, thereby restricting molecular mobility and increasing the internal resistance of the formulation to flow under shear. In addition, the high density of hydroxyl groups (–OH) and aromatic rings promotes hydrogen bonding and π–π interactions, thereby increasing the internal cohesion of the bio-binder.

Glycerol, in turn, is characterized by relatively high intrinsic viscosity (approximately 1.4 Pa·s at room temperature). Moreover, its three hydroxyl groups enable the formation of an extensive three-dimensional hydrogen-bonding network with surrounding molecules, including lignin and furfural. Consequently, increasing the glycerol proportion may increase the density of this cohesive network, thereby enhancing internal friction within the medium and increasing the overall viscosity of the bio-binder.

Finally, furfural is a reactive aldehyde. Under acidic conditions, which are favored by the relatively low pH of the formulation, and at elevated temperature (80 °C), it can participate in condensation and resinification reactions. Furfural may react with lignin-derived phenolic functionalities and other reactive hydroxyl-containing species, including glycerol, leading to the formation of oligomeric structures and, depending on the reaction pathway, acetal-type linkages. Even before the system reaches the gel point, the progressive formation of increasingly branched and larger molecular species increases the hydrodynamic volume and the apparent molecular weight of the system, thereby contributing to the marked increase in viscosity.

4.2.3. Effect of lignin, furfural, and glycerol on gel time

The results presented in Figure 3c show that lignin concentration has a practically negligible effect on the gel time, in contrast to the marked effects observed for furfural and glycerol. This difference in behavior can be explained by the distinction between the chemical kinetics of crosslinking, which are mainly governed by small reactive molecules, and the structural role of the macromolecular component. Furfural is a small and reactive unsaturated aldehyde that acts in the present system as a major crosslinking agent. Its relatively high molecular mobility facilitates its diffusion within the formulation and its interaction with reactive functional groups. Thus, increasing the furfural concentration increases the availability of reactive aldehyde groups and the frequency of reactive encounters, thereby promoting network formation and reducing the time required to reach the gel point.

Similarly, glycerol contains three readily accessible hydroxyl groups that can participate in condensation reactions under the conditions of the formulation. Changes in its concentration may therefore modify the effective stoichiometric balance of reactive groups and consequently affect the rate at which the system approaches the gel point (Dongre et al. 2015; Bai et al. 2025).

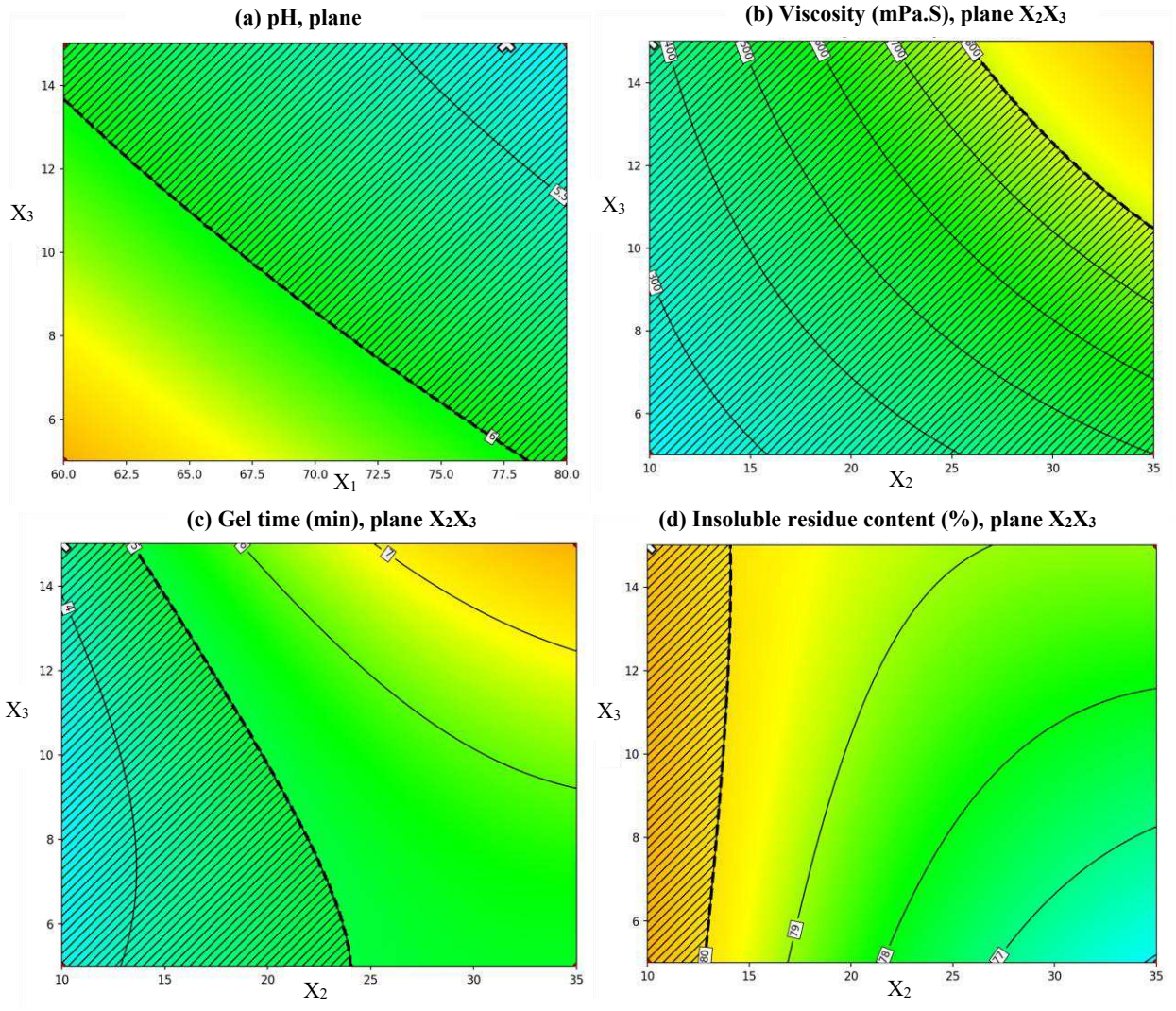
In contrast, lignin is a high-molecular-weight, highly branched macromolecule whose reactive functional groups may be less accessible because of steric hindrance and restricted molecular mobility. Increasing the lignin content therefore primarily introduces larger pre-existing macromolecular structures into the formulation. Although this increases the viscosity and potentially the rigidity of the system, as observed above, it does not necessarily result in a proportional increase in the number of kinetically accessible sites capable of accelerating gel formation within the concentration range investigated.

Overall, these results suggest that furfural and glycerol play a more prominent role in determining the gelation kinetics, whereas lignin appears to contribute mainly to the rheological and structural characteristics of the bio-binder. The negligible effect of lignin on gel time within the investigated range therefore indicates that its concentration primarily influences the physical structure of the formulation rather than acting as the dominant kinetic driver of gelation (Dongre & Bujanovic, 2021).

4.3. Modeling of the bio-binder formulation

The results presented in Tables 11 and 12 show that the mathematical model (Table 10) is statistically validated, indicating that its responses (Y_{pH}, Y_{Visco}, Y_{GT}, and Y_{IR}) can be optimized. The mathematical models for Y_{pH}, Y_{Visco}, Y_{GT}, and Y_{IR} reveal various effects of the interactions between the factors on the properties of the bio-binder. Thus, X_1X_3 was the interaction with the

greatest positive effect on pH, whereas X_2X_3 was the interaction with the greatest negative effect on gel time and the greatest positive effects on viscosity and insoluble residue content. These results indicate that glycerol (X_3) is the factor whose influence is most pronounced when combined with another factor through interaction effects. The response surface plots illustrating the effects of these interactions (X_1X_3 and X_2X_3) on the different responses are presented in Figure 5.

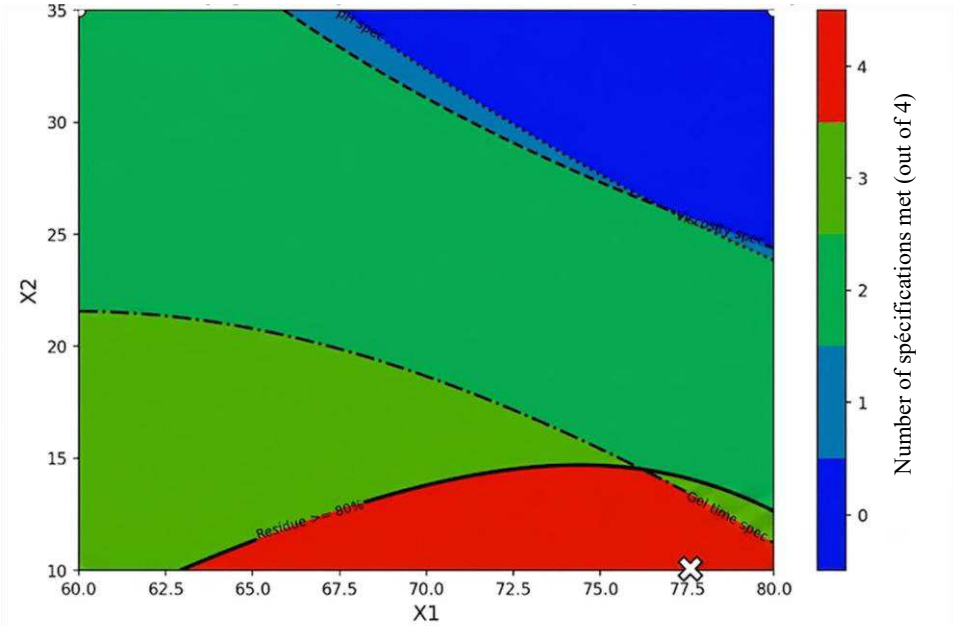


X_1 = Lignin ; X_2 = Furfural ; X_3 = Glycerol

Figure 5: Influence of interactions on pH, viscosity, gel time, and insoluble content of the bio-binder produced

Figure 5a shows that a bio-binder with a satisfactory pH (< 6) can be obtained with a lignin proportion > 77.5% and a glycerol proportion ≥ 13.5%. By fixing the lignin proportion at 77.6%, Figures 5(b, c, and d) show that, to obtain a bio-binder with a viscosity ≥ 60 mPa·s, a

gel time < 3.5 min, and an insoluble residue content > 80%, it is necessary to incorporate at least 13% furfural. However, to achieve optimal values for each factor while satisfying the different response criteria, a compromise must be found among the optimal regions identified in Figures 5a-d. This compromise was determined by superimposing the response surfaces shown in Figure 5. Figure 6 shows the result of this superposition in the search for the optimal point proposed by the compromise.



Red zone: all 4 specifications are met at the same time (10.8% of the domain).

Selected solution: 77.6% lignin, 10.0% furfural, 15% glycerol

(overall desirability D = 0.799).

X_2 = Furfural ; X_3 = Glycerol

Figure 6: The optimal zone (red) resulting from the superposition of the iso-responses of the different responses.

Figure 6 shows that the bio-binder formulation will be optimized by using a composition of 77.6% lignin, 10.0% furfural, and 15.0% glycerol. These values were tested in a bio-binder formulation trial, and the results are presented in Table 16.

Table 16: Experimental validation of the optimal values obtained

Response	Predicted Values	Experimental Values	Reference
pH	5.40	5.10 ± 0.20	5.0 – 6.0 (ASTM E70-19)
Viscosity (mPa.s)	378	342 ± 28	200 - 800 (ASTM D1084-16)

Gel-time (min)	4.27	3.35 ± 0.16	2 - 5 (ASTM D3056-14)
Insoluble residu (%)	80.55	79.56 ± 0.81	≥ 80 (Umemura et al., 2013)

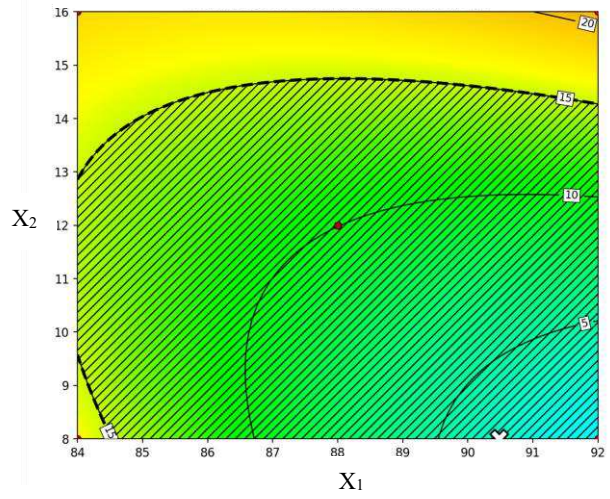
Optimum points : 77.6% lignin / 10.0% furfural / 15.0% glycerol

The experimental values obtained meet the specifications set at the outset. This validates the results obtained and their application in the test for manufacturing a compacted particle board.

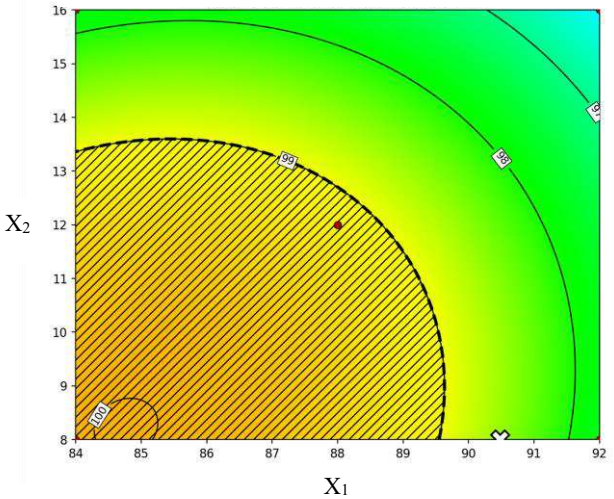
4.4. Modeling of the compacted particleboard based on the formulated bio-binder

The results presented in Tables 14 and 15 show that the mathematical model (Table 13), developed from the experimental trials for the production of particleboard using the formulated bio-binder, is statistically valid, and that its responses can therefore be optimized. The mathematical models for Y_{TS}, Y_{IR}, and Y_{AW} indicate the presence of an interaction between the two factors (X_1X_2) in each equation, affecting the properties of the compacted particleboard produced. This interaction is relatively weak and, specifically, is not statistically significant for either the impact resistance or the water absorption of the compacted particleboard. The response surface plots presented in Figure 7 identify the regions in which the combination of the two interacting factors could yield particleboards with low thickness swelling, high impact resistance, and low water absorption.

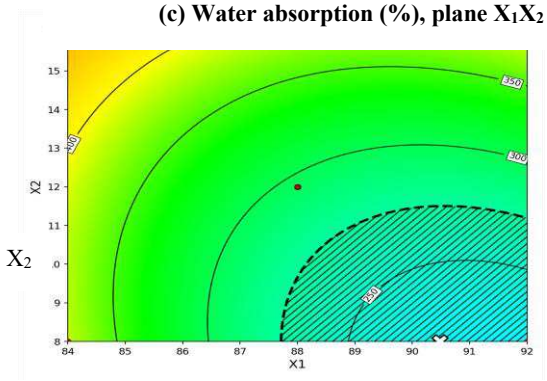
(a) Thickness swelling (%), plane X_1X_2



(a) Impact resistance (%), plane X_1X_2



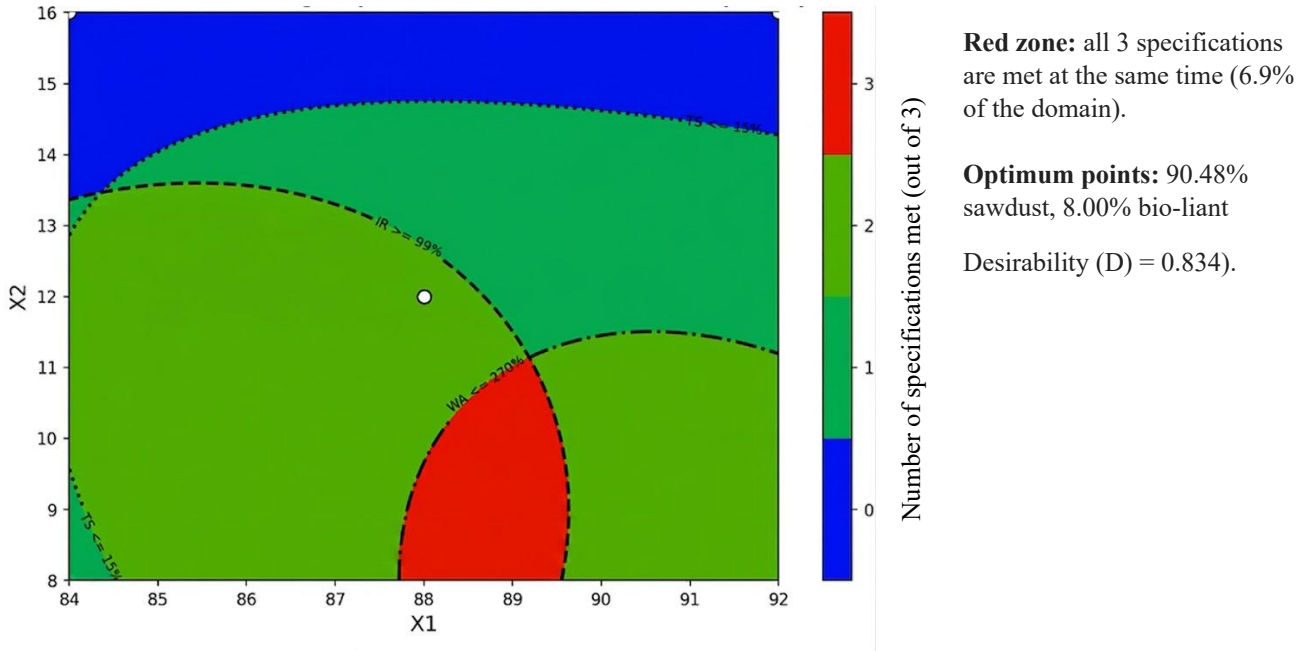
(c) Water absorption (%), plane X_1X_2



X_1 = Sawdust ; X_2 = Bio bander

Figure 7: Iso-response curves showing the areas with optimal values for obtaining a panel with low swelling (a), high impact resistance (b) and low water absorption capacity (b)

According to Figure 7a, to minimize swelling of the compressed particle board, it should be manufactured with a binder content between 9.5% and 13%, combined with a sawdust content greater than 84%. According to Figure 7b, to maximize the impact resistance of the compressed particle board, a binder content of less than 13.5% should be combined with a maximum of 89.5% sawdust. Finally, according to Figure 7c, a binder content of up to 11% should be combined with a minimum of 87.8% sawdust to minimize the water absorption rate of the compressed particle board. However, to obtain an optimal value for each factor in favor of the different responses, it was necessary to superimpose the iso-responses from Figure 7. Figure 8 shows the result of this superposition in the search for the optimal point offered by the compromise.



X_1 = Sawdust ; X_2 = Bio bander

Figure 8: The optimal zone (red) resulting from the superposition of the iso-responses of the different responses.

Figure 8 shows that the production of the compacted particleboard will be optimized by using a composition of 90.48% sawdust and 8.00% bio-binder as applied in this study. These values

were tested in a compacted particleboard production trial, and the results are presented in Table 17.

Table 17: Experimental validation of the optimal values obtained

Response	Predicted Values	Experimental Values	Reference
Thickness swelling (%)	3.71	2.94 ± 0,14	≤ 15 (EN 317, use dry)
Impact resistance (%)	98.57	100 ± 0.42	Maximum sought (relativ)
Absorption water (%)	234.59	254.24 ± 1,72	≤ 270

The experimental values obtained meet the initial specifications and validate the results obtained in this work. These values characterize the particle panel shown in Figure 9a.

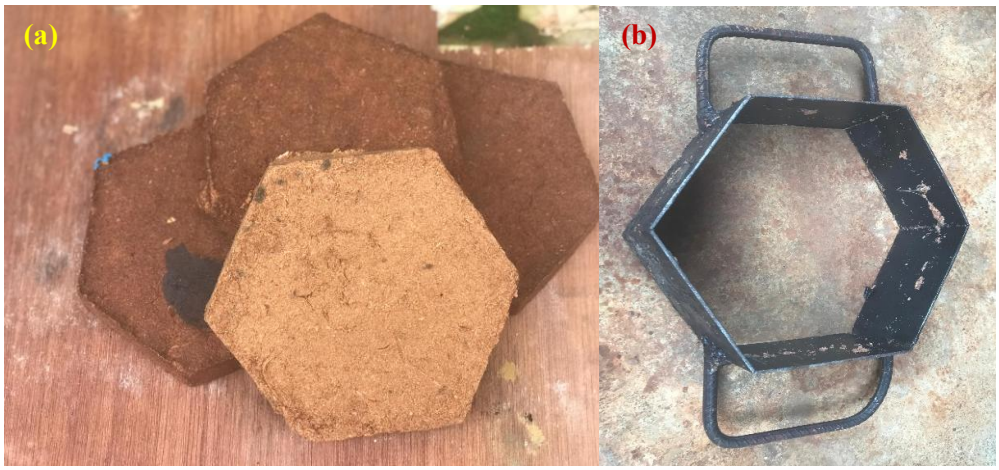


Figure 9: Image of compacted particleboard (a) made with a bio-binder, and its mold (b)

It should be noted that, with the exception of glycerol, all the inputs used to produce the bio-binder come from mixed sawdust of local wood species. Indeed, the lignin used was obviously extracted from this mixed sawdust. Similarly, the furfural and phenol used were extracted from the hemicellulose and lignin contained in this mixed sawdust, respectively. Ultimately, since the bio-binder is composed of 87.6% sawdust, its incorporation at 8.00% in the manufacture of the compacted particleboard (which contains 90.48% sawdust) makes this product a material

composed of 97.28% sawdust. This demonstrates the economic and industrial potential of this abundant and readily available waste material in Cameroon.

5. Conclusion

This study was out to develop a bio-based binder from composite sawdust derived from three local wood species. Overall, the results show that blending sawdust from different wood species can increase the lignin content of the composite feedstock. The lignin extracted through the Organosolv process exhibited a purity of approximately 91% and a solubility of 99% in the organic solvent. Phenolation of the extracted lignin increased its phenolic hydroxyl content by 38.72%, thereby enhancing the availability of reactive sites for subsequent crosslinking with furfural during bio-binder formulation.

The mathematical models generated from the experimental design showed that increasing the three formulation factors—lignin, furfural, and glycerol—significantly decreased the pH while increasing the viscosity of the bio-binder. The results further indicated that furfural and glycerol were the main factors governing the reaction rate and, consequently, the gel time, whereas lignin primarily influenced the final rheological and mechanical properties, particularly viscosity and rigidity, without being the main kinetic driver of gelation within the concentration ranges investigated.

An optimized bio-binder meeting the predefined conventional specifications was obtained using 77.6% modified lignin, 10.0% furfural, and 15.0% glycerol. This bio-binder was successfully incorporated at 8.0% into a formulation containing 90.48% composite sawdust to produce a compacted particleboard. Considering that the developed bio-binder itself contained 87.6% wood-derived material, its incorporation at 8.0% into the particleboard formulation resulted in a final material containing approximately 97.28% wood-derived material. These findings highlight the considerable potential of abundant and locally available wood-processing residues in Cameroon as a renewable raw material for the development of high-bio-based-content binders and value-added engineered wood products.

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Author contributions

Xavier Hyacinthe Wantouna Nduimo: Methodology, Soltware, Investigation, Writing-Original Draft**. Michael Bong Alang :** Formal analysis, Data Curation, writing (review and editing). **Esther Maurelle Ngouyamnsa Ghenmi:** Methodology, Investigation, Formal analysis, Data Curation. **Eric Edmond Fagsseu:** Conceptualization, Soltware, Formal analysis, Writing-Review and Editing**. Marie Josée Mafpouet Tionda :** Methodology, Soltware, Investigation. **Thierry Nguemtué Nguemtué** : Investigation, Formal analysis. **Eric Noubissie:** Conceptualization, Méthodology, Soltware, Formal analysis, Data Curation, Writing-Original Draft, Resources, Visualization, Project administration, supervision, Writing-Review and Editing. **Bertrand Charrier**: supervision, writing (review and editing). All authors have read and agreed to the published version of the manuscript.

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Data Availability

Data are available from the corresponding author upon reasonable request. A process of technology transfer is mandatory for any organization that is interested in using data for particular purposes.

Conflicts of Interest

The authors declare no conflict of interest.

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