Chapter 3
Experimental Characterization of Structural Elements Based on Polylactic Acid and Cocoa Pods of Cameroonian Origin

Audrey Tchamen Ngoule, Rostand Moutou Pitti, Loic Chrislin Nguedjio, Blaise Mtopi Fotso, and Cédric Delattre

Abstract This study investigates the valorization of cocoa pod husk powder (CPHP), an abundant agro-industrial by-product in Cameroon, as a bio-based reinforcement in a polylactic acid (PLA) matrix for the development of sustainable biocomposites. The composites were prepared by twin-screw extrusion with filler contents ranging from 10% to 30%, followed by thermal compression to produce films. Mechanical properties were evaluated through tensile testing. The results indicate that the incorporation of CPHP increases the rigidity of PLA-based materials, with an optimal compromise observed at 20 % filler content, where tensile strength (60 MPa) remains close to that of neat PLA. However, higher filler loading (30 %) leads to a marked reduction in tensile strength and ductility due to poor interfacial adhesion between CPHP particles and the PLA matrix. The rigid particles act as stress concentrators, limiting stress transfer efficiency and promoting crack initiation under tensile loading. In addition, composite films exhibited demoulding difficulties attributed to insufficient interfacial cohesion and the absence of release agents. Overall, this work demonstrates the technical feasibility of transforming cocoa pod husk waste into bio-based composite materials. The findings highlight the importance of interfacial optimization to enhance mechanical performance and support the development of sustainable materials for packaging and semi-structural applications.

Keywords Cocoa pod husk · PLA · Twin-screw extrusion process · Biocomposites

Introduction

The agri-food industry is experiencing strong global growth, particularly in sub-Saharan Africa [1]., but it generates large quantities of agro-industrial waste, creating significant environmental challenges [2]. Among the most polluting sectors is cocoa production, whose increasing output leads to the massive accumulation of cocoa pods. In Cameroon, one of Africa's leading producers with over 280,000 tons annually, each ton of dry cocoa beans generates approximately 10 tons of fresh pods. In the absence of structured recovery systems, these residues are generally left in the fields or burned, resulting in greenhouse gas emissions, air pollution, and soil degradation.

They can be used to produce potash for traditional soap manufacturing [3], as well as activated carbon with valuable adsorption properties. Therefore, transforming these agricultural residues represents a major environmental and economic opportunity for cocoa-producing countries.Plastic remains one of the major modern environmental pollutants, representing about 10% of household waste, much of which ends up in landfills or the natural environment. Poor waste management,

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particularly incineration, releases toxic substances that pose serious health risks. In response, the development of bio-based and biodegradable alternatives has become essential. Polylactic acid (PLA), a plant-derived thermoplastic produced from renewable resources such as corn starch or sugarcane, has emerged as a promising solution due to its biodegradability and good mechanical properties. However, its brittleness, low impact resistance, and thermal sensitivity limit its use in demanding applications such as construction. To address these limitations, research has focused on reinforcing PLA with plant fibers to enhance its mechanical and environmental performance, supporting the broader goal of building a more sustainable society. [4]. Cocoa pod residues, rich in cellulose, hemicellulose, and lignin, have properties suitable for reinforcing biodegradable polymers, similar to other plant fibers like hemp or flax [5]. However, specific research on the use of cocoa pods as a natural filler in PLA remains very limited [6, 7]. Our work fits precisely into this perspective, seeking to fill this knowledge gap and propose a path for local valorization, with our work already confirming the potential of this resource [8].

Material and methods

Cocoa Pods

The cocoa pods used in this study were collected in March 2025 in the town of Mbanga, located in the Mungo division of the Littoral region of Cameroon (Fig. 1). This area is known for its high cocoa production and is one of the country's main producing regions. The cocoa industry is a key sector of the local and national economy, generating employment for several million people [9].

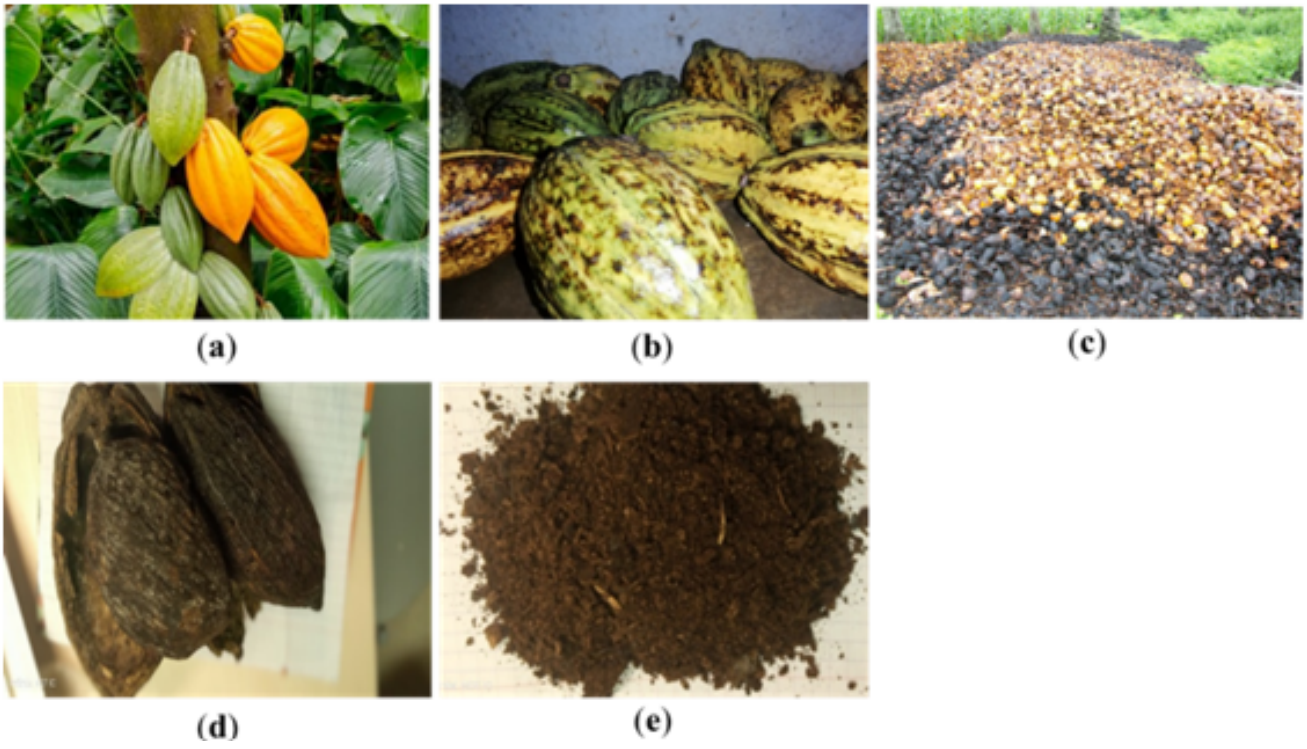

Fig. 1 (a) Cocoa plantation, (b) cocoa 5 days after harvest, (c) cocoa waste, (d) dried cocoa pods, (e) cocoa pod powder.

Polylactic acid

The PLA used in this study is Ingeo Natureworks 4043D, a biodegradable thermoplastic derived from renewable resources. It is widely used in composites due to good mechanical properties and processability, with a melting point around 170°C and molecular weight suitable for extrusion and injection molding. Its properties depend on molecular structure, crystallinity,

crystal size, and polymer chain arrangement, and it can be semi-crystalline or amorphous depending on monomer stereochemistry [10].

Composite preparation

Before any treatment, a preliminary observation was carried out on the cocoa. It was found that, without specific treatment, these shells begin to degrade naturally after about 14 days. After collection, they were dried at 100°C to remove moisture, then ground and sieved to a particle size of less than 200 microns, promoting better dispersion in the composite matrix and improving their mechanical performance. In this study, several composites were formulated by varying the proportions of CPHP and PLA: 100% PLA, 90% PLA – 10% CPHP, 80% PLA – 20% CPHP, and 70% PLA – 30% CPHP. The mixture was produced by twin-screw extrusion (HAAKE Minilab II) at 170°C and 100 rpm, with a continuous feed of approximately 6 g of material (Fig. 2). This method ensured good dispersion of the filler in the matrix and effective homogenization of the composites.

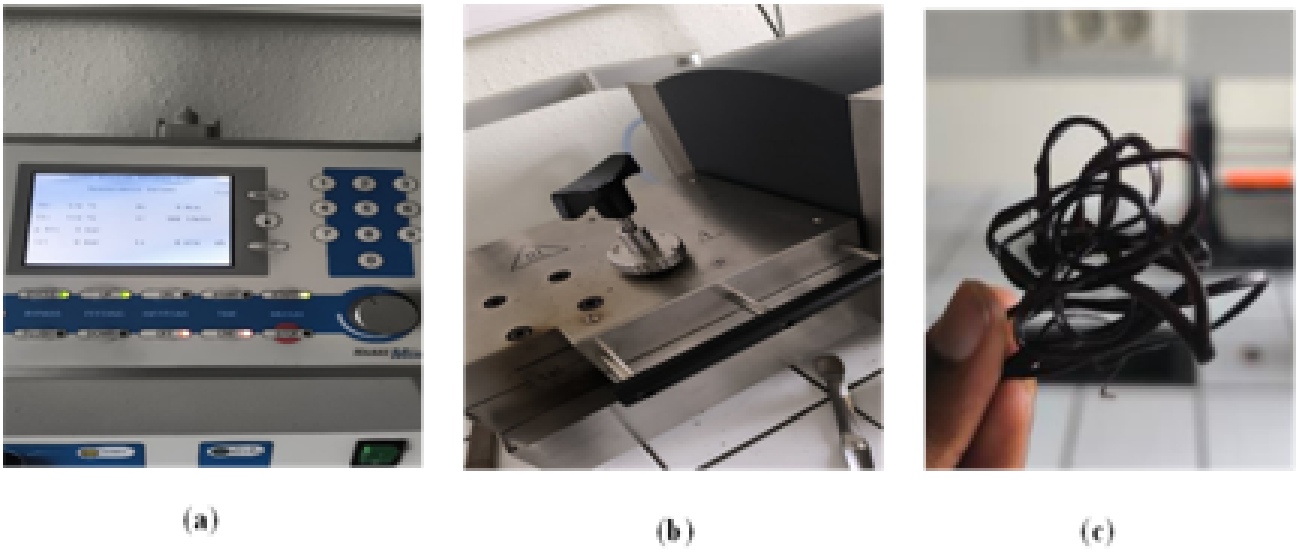

Fig. 2 Twin-screw extrusion (HAAKE Minilab II): (a) Control panel; (b) Hopper; (c) Extrudates.

Manufacture of composite films

The films were manufactured from extrudates and pure PLA by thermal compression using a Fontijne ICCF0647 press. Two masses (0.5 g and 1 g) were tested to evaluate the effect on the thickness and quality of the films. The material was placed between two metal plates covered with non-stick mats, then subjected to 180°C and 200 bar: 30 seconds of preheating followed by 60 seconds under pressure. After cooling to room temperature, the films were removed from the mould for characterization. The thickness was measured using a POPPI MI20 dimensional machine, taking several measurements per film to calculate the average. The incorporation of plasticizers into PLA makes it possible to produce flexible and compostable films suitable for sustainable food packaging applications.

Film tensile testing

The test specimens were cut from the previously manufactured films using a Druckleistung HK 800 manual press (capacity 8 KN, brand Best-Nr 30045, reference FUTURO). This knee lever press, equipped with flat jaws, ensures uniform pressure across the entire thickness of the films. In accordance with standard NF-EN-ISO 527-3, the samples have a length between jaws of 50 mm and a width of 15 mm, dimensions suitable for plastic films. Tensile tests were performed on an LS1 universal testing machine (AMETEK, Lloyd Instruments), equipped with a 100 N force sensor, allowing precise measurement of the low mechanical resistance typical of films. The system, controlled by NEXYGENPlus software, automatically records force, displacement and time with a displacement resolution of less than 1 μm (Fig. 3).

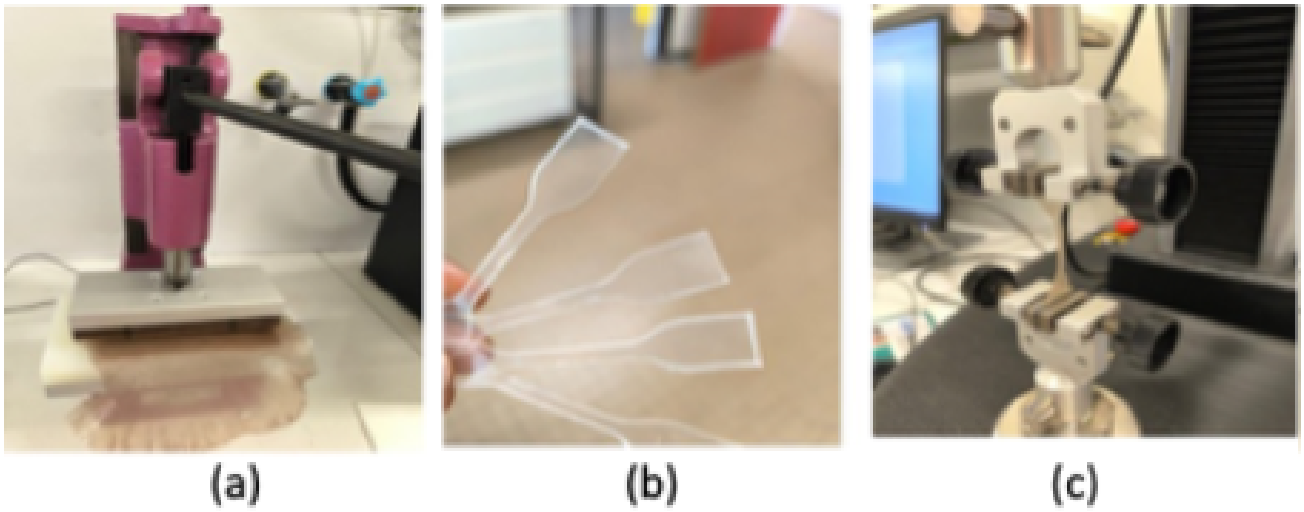

Fig. 3 (a) Cutting specimens on an HK800 machine; (b) Tensile test pieces; (c) LS1-AMETEK tensile compression testing machine.

Results and discussions

Composite films

Composite films based on cocoa shell powder (CSP) and polylactic acid (PLA) were obtained by thermal compression. After cooling, they were difficult to remove from the mould, as the CSP/PLA films adhered strongly to the heating plate, causing breakage, tearing or deformation (Fig. 4.b and 4.c). In contrast, films made of pure PLA or with a high proportion of PLA detached easily without damage (Figs. 4.a and 4.d). This difficulty is related to the low cohesion between the PCC particles and the PLA matrix, as well as the absence of release additives. A higher PLA content improves the continuity and homogeneity of the polymer network, strengthening internal cohesion and facilitating film removal.

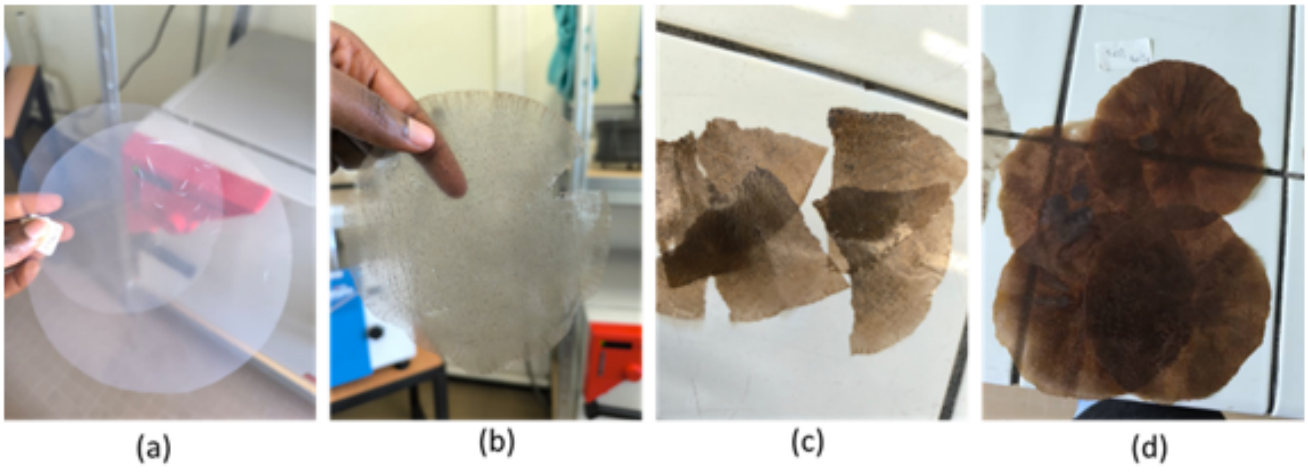

Fig. 4 Film manufacturing steps: (a) 100% PLA; (b) 90% PLA and 10% PCC; (c) 80% PLA ; (d) 1g of 90%PLA+10%PCC.

Tensile testing of films

The incorporation of CPHP into PLA films leads to a significant decrease in their tensile strength. This decrease is due to the presence of rigid particles that create defects and partial adhesion to the matrix, thus weakening the cohesion of the material under stress (Table 1).

Table 1 Film tensile tests.

	Force (N)	Stress (Mpa)	Strain (%)
Pure PLA 4043D	22.99	66.98	5.88
PLA Pure 4043D+ Cocoa pod husk powder	2.50	2.74	3.00

This quantitative observation corroborates the mechanistic explanation that cocoa pod husk powder (CPHP), acting as a rigid filler, introduces major structural defects into the material. The drop is directly related to the low interfacial adhesion or partial adhesion between the CPHP surface and the PLA matrix. This lack of compatibility results in low cohesion of the composite material. The rigid particles create stress concentration zones and defects that weaken the material, facilitating the initiation and propagation of cracks under tensile stress. The efficiency of stress transfer between the matrix and the filler is therefore severely compromised, resulting in the observed decrease in tensile strength. This trend is consistent with observations made for other natural fibers incorporated into biodegradable polymer matrices. For example, studies on wood, banana, bamboo, and sisal fibers also show that the addition of lignocellulosic fillers can increase the stiffness of the material but often reduces tensile strength and ductility, especially when the fibers are not pre-treated to improve their compatibility with the matrix [11]. In the case of PLA/CPHP films, the high embrittlement can be explained by the small size and stiffness of the particles, which generate stress concentration zones, as well as low interfacial adhesion. In the case of wood fibers in PLA, tensile strength decreases moderately with 10–20% filler, but stiffness increases slightly. The composites retain acceptable ductility [11]. For banana or bamboo fibers, the improvement in stiffness is often accompanied by a significant decrease in strength and elongation at break if the fibers are not chemically treated [11]. Another study developed biodegradable plastic films from agricultural waste, including cocoa pods and sugar cane bagasse, by extracting cellulose and fiber from these materials. Films containing 75% cellulose and 25% fiber exhibited the best properties for food packaging, with the lowest water absorption and water vapour permeability [12]. These comparisons confirm that, despite the potential for plant fibers to stiffen, optimising the fiber/matrix interface is very important for preserving mechanical strength, particularly for thin films where local stress concentrations are more critical.

Conclusion and future work

This study demonstrated the feasibility of valorizing cocoa pod husk powder (CPHP) as a bio-based reinforcement in a polylactic acid (PLA) matrix. Biocomposites were prepared by twin-screw extrusion and subsequently processed into films by thermal compression, with filler contents ranging from 10 to 30%. Mechanical testing showed that the incorporation of CPHP increases the stiffness of the material, with an optimal compromise at 20% filler, where tensile strength (60 MPa) remained close to that of neat PLA. At higher filler loading (30%), a significant decrease in tensile strength and ductility was observed due to poor interfacial adhesion between CPHP particles and the polymer matrix. The rigid particles acted as stress concentrators, limiting effective stress transfer and promoting crack initiation. The composite films also exhibited demoulding difficulties, attributed to insufficient internal cohesion and the absence of release agents, especially at higher filler contents. Overall, this study confirms the potential of cocoa pod husks for producing bio-based composites. Improving the filler/matrix interface through surface treatments or compatibilizers is essential to enhance mechanical performance. These findings support the development of sustainable materials from local agricultural residues, contributing to a circular economy and reducing agro-industrial waste. Future research should investigate the life cycle assessment of CPHP/PLA biocomposites to quantify their environmental benefits compared to conventional petroleum-based plastics and neat PLA. Evaluating parameters such as carbon footprint, energy consumption, and end-of-life scenarios would provide a comprehensive understanding of their sustainability potential and support their industrial-scale implementation.

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