Chapter

Mechanical Properties and Chemical Stability of Bathroom Wall Composites Manufactured from Recycle Polyethylene Terephthalate (PET) Mixed with Cocoa Hull Powder

Paul Nestor Djomou Djonga, Ahmat Tom, Hambate Gomdje Valery and Georges Elambo Nkeng

Abstract

The recovery of plastic waste and agricultural residues has led us to develop composites based on polyethylene terephthalate (PET) filled with cocoa shell powder. These shells have been previously treated with the organosolv process to improve the fiber-matrix interaction. The objective of this work is to develop wall covering materials to replace tiles which require a lot of energy and from PET. The composites were made by the method of melt mixing followed by compression molding. The mechanical, physico-chemical properties and stability to environmental conditions were evaluated. The results showed that the incorporation of 20–30% of powder in the matrix made of PET gave rise to a composite material with good properties for application in construction, as a wall covering replacing the tile. The study showed that the optimum powder weight ratio for optimum composite properties was achieved at a powder weight ratio of 30%. The maximum tensile strength of 60.3 MPa, bending strength of 19.5 MPa, impact strength of 10.3 MPa and water absorption of 1.34% were obtained. Compared with ceramic tile, this water absorption test value is within the range and shows that this composite tile is suitable for use as a bathroom tile.

Keywords: chemical stability, cocoa hull powder, composites, mechanical properties, PET

1. Introduction

Plastic consumption and its latter disposal have become a problem due to the high volume of waste and the huge environmental impact they have, not only for the human population, but also for ecological systems [1–4]. Plastic is a versatile material with wide applications. However, it is a material that people do not consume correctly as there are no perceived dimensions on the environmental damage that its use entails

[1, 5, 6]. Per capita plastic consumption continues to rise and remains high in high-income countries, despite obvious contributions to the global issue of plastic pollution [1, 7]. In 2015, the World Bank concluded that if waste generation maintains the same dynamic without adequate actions to improve reuse, unsustainable use, and production, it will have become a health emergency issue in most countries. This is in addition to high greenhouse gas emissions by 2030. Our planet is not capable of digesting the plastic waste generated daily, which will continue to happen. In Cameroun precisely in Yaoundé and Douala town, each person consumes an average of 2 kg of plastic per month, 24 kg per year, which means 1250 Mt. per year in those town [8]. The propagation of plastic waste in the environment constitutes a serious threat to public health because it contains in their structures pollutants and heavy metals that have enormous consequences on the health of living beings [9–11]. With the increase in the volume of waste in our cities, it is more than urgent to consider means of reducing these volumes. As a result, while most of the plastic available today is made from non-biodegradable sources, landfilling using plastic would mean burying the harmful material for a period of time until it naturally degrades. However, their degradation rate and bulky nature create enormous environmental risks. In addition, the mass of plastic waste can hinder the movement of groundwater [12], hence the need to give new life to the waste that would constitute a raw material for the construction industry of building materials. Plastic waste can be reused in several sectors of life. Several processes are used. We will cite thermochemical and thermal processes. A very interesting way to dispose of plastic waste is mechanical recycling, which consists of collection, shredding, and granulation followed by its reintroduction into the manufacture of other plastic products [13]. Given the soaring costs of construction materials, the ambient unemployment that many young people suffer, this valuation sector can create jobs and reduce poverty, which results in the valuation of local raw materials as construction materials. The valuation of plastics in construction materials finds many applications in civil engineering. This on the one hand to the economic advantages is provided by this material, which can act as a binder as a substitute for cement. In addition, several waste management processes are used in civil engineering and have shown that the substitution or use of this material could validly replace binders and would constitute an excellent binder but with low compressive strength values. Much work has been done on the recovery and capacity of plastic waste to be used as binders or as a replacement for cement, see the association of plastic cement in concrete. The most commonly used plastics are polyethylene terephthalate (PET) bottles, polyvinyl chloride (PVC) pipes, high-density polyethylene (HDPE), used plastic waste, expanded polystyrene (EPS) foam, reinforced plastic. Glass (GRP), polycarbonate, recycled thermoplastic polystyrene, polypropylene fiber act as aggregate or mixture in the manufacture of concrete [9].

Reused plastics can be used as reinforcement with natural or synthetic textile fibers in the manufacture of composite materials, the main application of which is the coating of surfaces, walls replacing tiles. Interest in natural fiber-reinforced composites (NFRC) is arising due to their properties of biodegradability, noncarcinogenicity, profitability, and respect for the environment, the absence of health risks, easy collection, and regional availability. They are also used as renewable resource, thus offering a better solution for the sustainability of supply [13]. The different properties of the NFRC show that they would find wide application in the automotive sector, railroad cars, building construction, wall partitions, cabinets, furniture, and packaging manufacturing [6]. NFRCs are viable due to the wide availability of natural fibers and fibers from agricultural residues or mostly lignocellulosic can be used as reinforcements [13, 14]. Several authors have embarked on the search for fibers or natural woody materials having properties similar to synthetic fibers from a physico-mechanical point of view that can be naturally or directly used as reinforcement in composites [13, 15].

Indeed, polymers based on synthetic fibers as reinforcements are expensive and have harmful consequences on the environment. Despite the advantages, natural powder has a high water absorption capacity, hydrophilic in nature, which makes it difficult to manufacture composites. The powder swell is softened when in contact with humidity and consequently absorbs water, which contributes to the reduction of their mechanical properties, while its hydrophilic character affects the dispersion of the fibers within the matrix phase. To improve the interaction between the powder and the polymer matrix, natural fibers must be physically or chemically modified to increase their reactivity and their physico-mechanical properties. Much work has been done on composites reinforced with natural fibers such as kenaf, palm oil, bamboo, jute, sisal, coconut, and pineapple fibers. But there is hardly any work in the literature that uses cocoa shell powder as filler. Cameroon is at the forefront of the production and exportation of certain cash crops, such as cocoa (fifth largest producer in the world), bananas, pineapples. It has embarked on a process of modernizing its agriculture and is currently implementing the so-called second generation agriculture, which aims to boost national agricultural production. This generates a lot of agricultural waste. Thus, in 2013, around 700,368 tonnes of cocoa hulls were produced and around 2,900,000 tonnes will be produced by 2022 [16]. Only a small part has been used as fertilizer and animal feed [17], which poses environmental problems, because these cocoa shells therefore pollute the soil and the rivers. This research aims to use the powder from the cocoa shell to strengthen recycled PET. The tensile strength, bending, water absorption, and resistance to acidic and basic conditions will be evaluated in order to measure the physicochemical and mechanical properties of composites.

2. Materials and methods

2.1 Materials

The raw material used in this work consists mainly of waste plastics (PET) used as a binder and cocoa hull powder.

The waste plastic samples were taken from the Center region of Yaoundé, Cameroon. Plastic waste was collected in the Mendong area of Yaoundé. **Table 1** shows us the physical and mechanical characteristics of plastics (PET).

2.2 Methods

2.2.1 Procedure for obtaining cocoa shell powder

After podding, the cockles were cut up and collected in a polythene bag transported to the laboratory, and then dried in a Heraeus brand oven at 45° C until a constant mass is obtained. The dried slices were crushed using a mechanical grinder and sieved with a 1-mm sieve. The powders obtained were packaged in plastic bags and stored at room temperature (25 ± 2°C) until use.

Properties	Absorption rate (%)	Density	Resistance to traction (MPa)	Mass volume (kg/m^3)	Latent heat of fusion (J/g)	Thermal capacity J/kg °C
PET	0.0016	0.93	24	1395	115	1090

Table 1.
Physical, mechanical, and thermal characteristics of PETs.

2.2.2 Physicochemical characterization of cocoa shell powder

2.2.2.1 Water and dry matter content

The water content was determined using the method described by AFNOR (1982) reported by Bachmann et al. [18]. A mass of the fresh sample is dried at 105°C in an oven for 24 h until a constant weight is obtained.

2.2.2.2 Crude fiber content

The determination of the crude fiber content in our powders was determined using performed according to the method described by treating the sample to the boil with sulfuric acid followed by soda. The postharvest by-product obtained is then dried in an oven at 60°C for 24 h, calcined in an oven at 550°C for 4 h with a rate of rise in temperature of 10°C/min and weighed [19].

2.2.2.3 Lignin quantity

The lignin level was determined by the Klason method reported by Monties [20]. The method consists in observing the power of insolubility in an acidic solvent in a concentrated medium and dissolving all the constituents present in the material. The aim of this analysis was to obtain a raw material rich in woody constituent.

2.2.2.4 Reducing sugars quantity

The reducing sugar content was determined using the DNS (acide 2-hydroxy-3, 5-dinitrobenzoïque) method described by Fischer *et al*. [8]. It begins with an extraction of sugars in a hot acidic medium. In a hot alkaline medium, DNS reacts with reducing sugars and changes from its yellow-oxidized form to its orange-reduced form with a maximum absorption at 530 nm.

2.2.2.5 Determination of mass loss

The difference between the mass of the sample before and after the pretreatment represents the mass loss. The sample is dried at105°C before and after pretreatment until a constant mass is obtained.

2.2.2.6 Pretreatment of hulls by the organosolv process

It consists of a batch reactor with a total volume of 100 mL with the useful volume being 80 mL. The experimental protocol was inspired from that described by Nanfack et al. [21]. A mass of 4 g of cocoa shell is mixed with 80 ml of the pretreatment solvent (ethanol-water mixture 52:48 v/v) and loaded into the reactor. The latter is tightly sealed using silicone glue to ensure it is watertight. It is then put in an oven preheated to 200°C. The treatment is stopped by cooling under running water to room temperature. After cooling, the two solid and liquid phases are separated by filtration. The cocoa shell powder thus treated is dried and stored in a glass jar for later use.

2.2.3 Manufacturing method of composite materials

In this study, fine-sized plastic waste is obtained after shredding with a chisel. Plastic waste is then washed with water and dried at room temperature (25°C) for 3 days.

% of CCP	0	10	20	30	40	50
% of PET	100	90	80	70	60	50

Table 2.
Formulation of the different composites according to the addition of the powder.

Composite for PET and cocoa powder: The appropriate with of cocoa powder was added to melt PET in a Haack Rheomix at 225°C. The mixture weighing 200 g was processed for 10 min. The mixture obtained was then hot press at 225°C for 5 min at a pressure of 15 MPa and cooled under pressure. The choice of the temperature of 225° C. was due to the fact that a preliminary sweeping was carried out in order to find the minimum temperature, which makes it possible to develop the composite so as not to alter the powder. From the work of the literature, the melting temperature of PET was around 250° C. Specimens of dimensions 200 × 150 × 8 mm were prepared using a parallelepiped mold. The mold has been polished with a mold release agent to prevent the PET from sticking to it. The process involved melting the grated PET, adding a predetermined proportion of cocoa powder, melt-mixing to form a homogeneous viscous solution, and placing into the prepared mold. Finally, the mold was closed and the samples were cooled to room temperature under a pressure of 15 MPa for 30 min. The specimens of the tiles produced were shaped by sandpaper and used for testing.

Sheets of 3 mm thickness were obtained. The materials were made at different powder concentrations as shown in **Table 2**.

2.3 Mechanical properties and chemical stability of composite

2.3.1 Tensile test

The tensile test evaluation was performed on the Instron 5567 with a climb speed of 50 mm/min. Preparation of samples for testing was performed according to ASTM D638. At the start of the analysis, the specimen lengthens and the resistance of the specimen increases, which was detected using a load cell; the value was recorded until the composite failed. For this, five samples were tested in order to evaluate the standard deviation [22–25].

2.3.2 Flexural test

Mechanical strength is the combination of tensile strength and compressive strength. The tests were carried out on a universal testing machine. The specimens were prepared according to the test standard ASTM D790 of dimension 200 × 30 × 8 mm. These samples of composites produced in varying proportions of PET were tested on a support span of 130 mm according to the standard [13].

2.3.3 Impact test

Charpy impact tests on notched specimens were performed using a model JBS-300 N pendulum impact testing machine. Specimens were prepared according to the ASTM D6110-18 test standard. With a dimension of 50 mm long and a cross section of 24 mm^2, five specimens were tested and one mean value was reported [13].

2.3.4 Water absorption

The water absorption test was performed on parallelepipedal specimens according to ASTM-C373 [22] where the composite samples were weighed and immersed

in a beaker containing distilled water and the whole boiled, for 2 h, and allowed to cool for 24 h at room temperature. The specimens were then wiped with a paper towel and weighed.

2.3.5 Chemical stability

The chemical stability will consist in soaking our composite tiles in an acid solution and a base solution with respective pH of 3 and 12 in order to evaluate the loss of mass over a period of 7 days.

3. Results and discussion

3.1 Characterization of cocoa powder hulls

3.1.1 Chemical composition of cocoa powder hulls

As shown in **Table 3** physicochemical properties of cocoa powder hulls is given below.

The dry matter content in our powder was of the order of 88.1%, against 92.1% in organic matter. This high organic matter content shows that cocoa hulls can be an important source of raw material for the production of composites given its organic matter content. The tensile test evaluation was performed on the Instron 5567 with a climb speed of 50 mm/min. Preparation of samples for testing was performed according to ASTM D638. At the start of the analysis, the specimen lengthens and the resistance of the specimen increases, which was detected using a load cell; the value was recorded until the composite failed. For this, five samples were tested in order to evaluate the standard deviation [13].

3.1.2 Thermal analysis

The analyses of the results of the thermal properties of the powder are presented in **Figure 1**. The thermogravimetric analysis (TGA) (in red) provides the loss of mass of the material during thermal degradation. The heat treatment process is that described by Verma et al. [26]. The blue-colored thermogravimetric (DTG) curve gives the shell degradation temperature profile as defined by Balakrishnan et al. [27].

Constituents	Before pretreatment
Dry matter content (g/100* g)	88.1 ± 0.1
Water content (g/100* g)	11.9 ± 0.1
organic matter content (g/100⁺ g)	92.1 ± 0.2
Ash content (g/100⁺ g)	7.9 ± 0.2
Reducing sugar content (g/100* g)	0.020 ± 0.003
Crude fiber content (g/100⁺ g)	40.1 ± 1.2
Lignin content (g/100⁺ g)	35.3 ± 1.8
Cellulose content (g/100⁺ g)	41.4 ± 0.3

*+—dry base, *—wet base.*

Table 3.
Chemical composition of cocoa powder hulls.

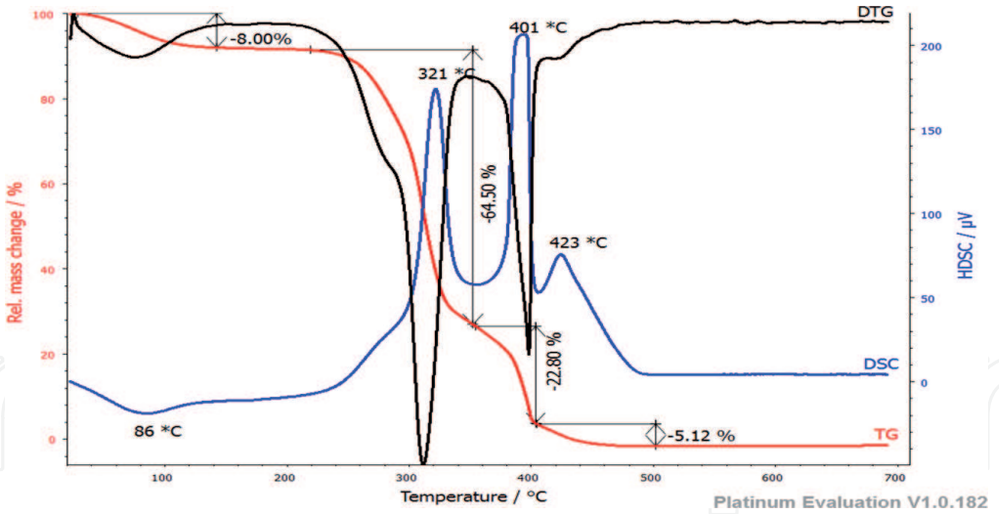

Figure 1.
Thermogravimetric and differential scanning calorimetry of raw cocoa powder hulls.

Careful observation shows that from 440°C all organic matters have been degraded.

The spectrum in **Figure 1** shows four main phases of mass loss. The first mass loss of 8.00% by weight is between 40 and 100°C and has a peak around 86°C; it reflects the loss of free water contained in the cocoa shell powder. The second loss of mass corresponds to a loss of mass of 64.50% observed between 300 and 400°C, with a maximum DTG at 321°C. It is attributed to the destruction of sugars, in particular polysaccharides (hemicelluloses and cellulose) in cocoa shell powder [26]. The third loss of 22.60% observed between 321 and 401°C could reflect the degradation of the cellulose contained in the powder; in fact, cellulose would degrade at around 401°C. Between 401°C and 423°C, a loss of 5.12% is observed with a peak at around 423°C, which would be due to the destruction of lignin at 423°C, these results are consistent with those found by [26].

With regard to the physicochemical properties of these powders, we can conclude that the major compounds are cellulose, hemicellulose, and lignin, which are present in our powder, they degrade with a strong loss of mass [28]. In addition, hemicelluloses decompose at low temperature, due to their weak molecular structure. In addition to their low molecular mass, they have less regular structures in their chains [28]. The curve in **Figure 1** shows four thermal accidents. One endothermic peak is observed at low temperature around 86°C and three exothermic peaks lie between 321°C, 401°C, and 423°C. The peak around 86°C would correspond to the free water contained in the powders, and the other peaks would reflect respectively the degradation of the hemicelluloses, celluloses, and lignin of the cocoa powder hulls [28]. **Table 4** shows the different types of thermal phenomena that occur during the thermal analysis of the sample.

	Temperature (°C)	Partial loss of mass (%)	Total loss of mass (%)	Type of reaction
Powder of cocoa hulls	86	8	100	Endothermic
	321	64.50		Exothermic
	401	22,8		Exothermic
	423	5.12		Exothermic

Table 4.
Types of thermal phenomena.

3.1.3 Infrared Fourier transform analysis

As shown in **Figure 2** and **Table 5**, the IRTF analysis of cocoa hulls powder shows different peak intensities.

Five main groups of adsorption bands emerge, namely those between 3000–3500, 3000–2500, 2000–1500, 1000–500 cm^{-1}. The wavenumber bands between 3000 cm^{-1} and 3500 cm^{-1} is attributed to the vibration of elongation of the linked alcohol OH group. The wavenumber between 2500 cm^{-1} and 3000 cm^{-1} shows the presence of the CH elongation bond. The adsorption band between 2000 cm^{-1} and 1400 cm^{-1} is the characteristic of CO elongation. The absorption band of 1000 cm^{-1} corresponds to the CC bond.

3.2 Mechanical characterization of composite materials

Different formulations were carried out with incorporation rates of the cocoa pod powder from 0 to 50%. The following properties, flexural strength, tensile

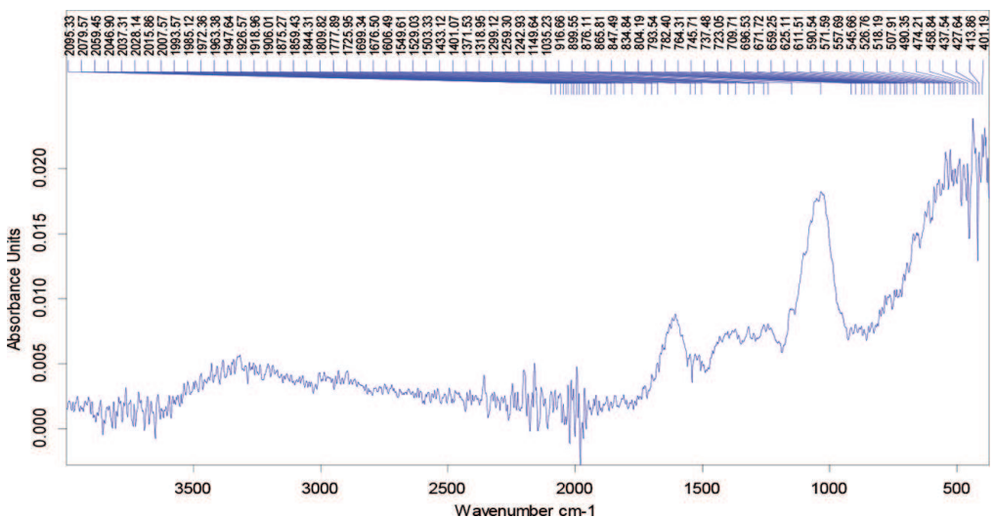

Figure 2.
Infrared of raw cocoa shell powder.

Number of waves (cm^{-1})	Attribution
3315	√ (OH) of lignin and polysaccharides
2927	$\sqrt{}_{as}$ (C–H) of CH and CH_2
1733	√ (C═O) esters and carboxylic acids (xylans, lignins)
1602	Lignin aromatic skeleton
1455	δ_{as} (C–H) from lignin and xylans
1417	δ_{as} (C–H) polysaccharides
1371	δ_{as} (C–H) of cellulose and hemicelluloses
1317	CH_2 from cellulose and hemicellulose
1234	√ (C–O) of methoxy groups of lignin
1025	δ (C–O) of polysaccharides δ (C–OH) of cellulose
896	δ (CH) of cellulose
600	δ (CO–H) of cellulose

√ is the strain in vibration and δ is the strain in rotation.

Table 5.
IR spectrum of cocoa shell powder: the characteristic infrared vibration bands, relating to CCP.

%CCP	Tensile strength (MPa)	Flexural strength (MPa)	Impact strength (MPa)	Water absorption (%)
10	55.8	10.6	8.9	1.18
20	58.2	11.9	7.4	1.20
30	60.3	19.5	10.3	1.34
40	56.1	12.3	7.6	1.49
50	43.9	9.8	6.9	1.51

Table 6.
Mechanical properties of composite.

strength, impact strength, and water absorption, have been used to define the properties of composites. As shown in **Table 6**, the mechanical properties of composites are given.

3.2.1 Tensile strength

The tensile strength was studied as a function of the rate of incorporation of the cocoa powder into the PET. As shown in **Table 6**, that the tensile strength of the composite increases with the amount of load introduced 10–30% and is equivalent to a value of 55.8–60.3 MPa, respectively, but when the level of 30% powder is reached, this value drops from 60.3 to 43.9 MPa. Observation of the table indicates that the greatest value of the tensile strength is reached after adding 30% of powder. This high value of the resistance results in a reinforcement of the PET, which has become more rigid and can withstand large loads. The small size and their particle size distribution in the composite allow good adhesion and contribute to the densification of the composite. Beyond 30% of the powder content, the resistance to decrease that would lead to believe that the PET matrix was not sufficient to cross and homogeneously fluidize the surface of the composite creating pores, which would contribute to the low resistance to traction.

3.2.2 Flexural property

Analysis of the table reveals that the flexural strength increases with the addition of the powder. The incorporation rate of 10 to 30% made it possible to go from a value of 10.5 to 19.50 (MPa). This could be explained by the fact that an increase in the loading rate would lead to the presence of a high rate of loading on a cross section of the composite and therefore increasing the flexural strength. We also note that beyond 30% of the incorporation rate, there is a decrease in the value of the flexural strength. This drop could be explained by poor dispersion of the powder in the matrix and this observation is confirmed by the tensile strength where we see similar results.

3.2.3 Impact strength

Table 6 presents the values of the Charpy impact resistance (CIS) of non-notched samples of PET composites with a proportion of cocoa shell powder varying from 10 to 50%.

For a load rate below 30%, there is an increase in the CIS, which reaches its highest value 10.3 (MPa) at 30% CCP. Beyond 30% the CIS decreases, because the addition of more powder creates regions of stress concentrations that require comparatively less energy to initiate a crack; this reduction could also be explained by the fact of the low resistance of the interface between the powder and the matrix. During impact, most of the energy absorbed is used to increase the distance between the load and the die.

Day	0	2	4	6	8	10
30% CCP at pH 3.2	150	143	141	139	139	138
30% CCP at pH 6.79	150	148	147	147	147	147
30% CCP at pH 12.4	150	151	151	150	150	150

Table 7.
Weight loss of specimen in nitric acid and sodium hydroxide solutions (g).

3.2.4 Water absorption

As shown in the **Table 6** the filler content has a notable effect on water absorption. After 24 h of immersion in water, the lowest water absorption rate was reached with a load proportion of 10%. The incorporation of cocoa shell powder would be help to increase the percentage of water absorption up to a certain content, which means that more hydrogen bonds are formed between the water molecules and the OH group present in the powders. Similar results were observed by Huner et al. who reported that the rate of water absorption increased with increasing fiber content [29, 30]. This could be explained by the formation of a less surface interaction between the matrix and the powder during mixing, resulting in a higher water absorption [31]. In addition, PET would constitute an ideal binder for the compaction of this powder and reduce water absorption. This low water absorption value could improve the physico-mechanical properties.

3.3 Chemical stability

As shown in **Table 7** that after 10 days of impregnation in acidic medium and neutral medium, the composite material undergoes a mass loss of 8 and 2%, respectively, whereas in a basic medium no loss is observed. This result reflects the good chemical stability of the composites under extreme conditions and shows that the material can indeed be applied as a tile coating for the walls of toilets and showers.

4. Conclusion

The chapter studied the chemical stability and mechanical properties of bathroom wall composites manufactured from recycle polyethylene terephthalate mixed with cocoa hull powder. Organosolv treatment of cocoa powder hulls enhanced the quality of the cocoa powder hulls by removal of hemicellulose and lignin thereby increasing the cellulose composition. The Organosolv treatment gave a more thermally stable composite material. The addition of cocoa powder hulls produces an environmentally friendly material. It is clear that cocoa powder hulls have great potential as a filler and reinforcement for composites requiring similar properties to PET. The recycled PET/30% cocoa hull powder blend has higher tensile, flexural, and impact strength compared to other blends. Recycled PET/30% cocoa hull powder blend was also found to have good properties in terms of stability. The manufactured composites showed improved mechanical properties with respect to the powder content increase, showing the reinforcement potential of cocoa powder. It is thus possible to consider a prospective industrial use of this agricultural waste, for instance, for the manufacture of bathroom wall tiles in construction industry in Cameroon.

Conflicts of interest

The authors declare that they have no conflicts of interest.

Data availability

All relevant data to the manuscript have been included.

Author details

Paul Nestor Djomou Djonga[1*], Ahmat Tom[2], Hambate Gomdje Valery[3] and Georges Elambo Nkeng[4,5]

1 Faculty of Science, Department of Chemistry, University of Maroua, Maroua, Cameroon

2 Department of Energy Engineering, University Institute of Technology, University of Ngaoundere, Ngaoundere, Cameroon

3 Departement of Textile and Leather Engineering, National Advanced School of Engineering of Maroua, University of Maroua, Maroua, Cameroon

4 Faculty of Science, Department of Chemistry, University of Buea, Buea, Cameroon

5 Ecole Nationale Supérieure des Travaux Publics, Yaounde, Cameroon

*Address all correspondence to: djomoupaul@gmail.com

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