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

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ABSTRACT

The need to protect our environment by eliminating plastic waste as much as possible and by recycling waste from agricultural residue, has led us to formulate composites based on polyethylene terephthalate (PET) loaded with powder from the cocoa shell. The cocoa hulls were prior treated with organosolv process to improve the fiber-matrix interaction. This research is aimed at manufacturing composite wall tiles from recycled PET reinforced with cocoa hull powder (CCP). The composites were manufactured by the melt-mixing method followed by compression molding. The mechanical, physico-chemical properties and the stability to environmental conditions were evaluated. The results showed that the incorporation of cocoa powder at a content of 20-30% in the matrix consisting of PET gave rise to a composite material with good physico-mechanical and chemical properties suitable for use in several sectors. In the construction industry, in particular as wall covering as a replacement for tiles, these x from an economic point of view cost less and compared to clays which consumes enormous amount of energy for the elaboration of ceramics. The study showed that the optimum powder weight proportion for the optimal properties of the composite were achieved at 30% powder weight proportion. The maximum tensile strength of 60.3 MPa, flexural strength of 19.5 MPa, impact strength of 10.3 MPa and water absorption 1.34% were obtained. Water absorption of the tiles increased with the cocoa powder weight. Compare to the ceramic tile this value of water absorption test is in range and show that this composite tile is suitable for use as bathroom tile.

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, 2–4]. 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 and this 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, this 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 which 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 which 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. In terms of civil engineering application, recycling of plastic waste as cementitious based materials. This is due to its economic and ecological advantages which can substitute or replace certain portion of aggregate in concrete mix. Besides, some alteration method of plastic waste could be ideal candidate as lightweight concrete pavement with low strength application. As such, there are already a lot of published reports that have been studied concerning the ability of plastic waste mixed together with cementitious based mixtures such as polyethylene terephthalate (PET) bottle, polyvinyl chloride (PVC) pipe, high density polyethylene (HDPE), spent plastic waste, expanded polystyrene foam (EPS), glass reinforced plastic (GRP), polycarbonate, thermoplastic recycled polystyrene, polypropylene fiber as an aggregate, or mixture in the manufacturing 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 increasing due to their properties of biodegradability, non-carcinogenicity, profitability, and respect for the environment, absence of health risks, easy collection and regional availability. They are also a renewable resource, thus offering a better solution for the sustainability of supply [13]. The NFRC versatile characteristics make it suitable for automobiles, railway coach, building construction, wall partition, cabinets, furniture, and packaging manufacture [6]. NFRC are viable because of the wide availability of natural fibers and agriculture by-product fibers which can be used as

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reinforcements [13, 14]. Researchers have been working round the clock to find new sources of natural fibers that possess comparable physical and mechanical properties to synthetic fibers to be used in reinforcing of 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 and soften in contact with humidity and consequently absorb 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 using 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 a filler. Cameroon is at the forefront of the production and exportation of certain cash crops, such as cocoa (5th largest producer in the world), bananas, pineapples, etc. It has embarked on a process of modernizing its agriculture, and is currently implementing 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, resistance to acidic and basic conditions will be evaluated in order to measure the physicochemical and mechanical properties of composites.

2. Experimental Methods

2.1 Materials

The raw material used in this work consists mainly of waste plastics (PET) used as a binder and cocoa hulls powder. The waste plastic samples were taken in the Center region of Yaoundé, Cameroon. Plastic waste was collected in the Mendong area of Yaoundé. Table 1 gives the physical and mechanical characteristics of collected plastics (PET).

Table 1 Physical, mechanical and thermal characteristics of PETs

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

2.2 Procedure for Obtaining Cocoa Shell Powder

After podding, the cockles were cut up and collected in a polythene bag transported to the laboratory. 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.

2.3 Physicochemical Characterization of Cocoa Shell Powder

2.3.1 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 hours until a constant weight is obtained.

2.3.2 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 residue obtained is then dried, calcined and weighed [19].

2.3.3 Lignin Content

The lignin content was determined by the Klason method reported by Monties [20]. This so-called Klason lignin method uses the insolubility

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property of lignin in a concentrated acidic medium that hydrolyzes and dissolves all other constituents. Its objective is to obtain an essentially woody residue.

2.3.4 Reducing Sugars Content

The reducing sugars content was determined using the DNS method described by Fischer *et al.* (1961). 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.

2.3.5 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 at 105 °C before and after pretreatment until a constant mass is obtained.

2.3.6 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.

2.4 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, dried at room temperature (25 °C) for three days. 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 were processed for 10 minutes. The mixture obtained was then hot press at 225 °C for 5 minutes at a pressure of 15 MPa and cooled under pressure. The temperature of 225 °C was used because it does not affect the powder properties. Composite tiles of size 200 × 150 × 8 mm were prepared using a closed mold. The mold was polished with a release agent to avoid PET from sticking to it. The process involved melting of the shredded PET, adding predetermined proportion of the cocoa powder, melt-mixing thoroughly to form a homogeneous viscous solution, and placing it into the prepared mold. Finally, the mold was closed and the samples were cooled down to room temperature under 15 MPa pressure 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 (Table 2).

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

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

2.5 Mechanical Properties and Chemical Stability of Composite

2.5.1 Tensile Test

The determination of the tensile tests was performed on the Instron 5567 at a crosshead speed of 50 mm/min. Samples were prepared for this test according to the ASTM D638 test standard. At the start of the tensile test, the test piece lengthens and the resistance of the test piece increases, which was detected using a load cell. This value was recorded until the sample ruptured, and five samples were tested [13].

2.5.2 Flexural Test

Flexural strength is the combination of tensile strength and compressive strength. The tests were done on a universal testing machine. The specimens were prepared according to the ASTM D790 test standard with dimension 200 × 30 × 8 mm. The specimens were tested on a support span of 130 mm as per the standard [13].

2.5.3 Impact Test

Charpy impact tests on unnotched specimens were performed using a pendulum impact testing machine JBS-300 N model. The test specimens were prepared according to the ASTM D6110-18 test standard with a dimension of 50 mm long, and a cross-sectional area of 24 mm². Five specimens were tested and an average value was reported [13].

2.5.4 Water Absorption

Water absorption was carried out on cylindrical fired test specimens according to ASTM-C373 standard [22] where ceramic samples were weighed and immersed in a beaker containing distilled water and the ensemble boiled for 2 hours, and allowed to cool for 24 hours at room temperature. The specimens were then wiped with a paper towel and weighed.

2.5.5 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

The cocoa hulls powder studied has a dry matter content of 88.1% and an organic matter content of 92.1%. This high organic matter content shows that cocoa hulls can be an important source of nutrients for microorganisms in anaerobic digestion, this can explain why it's used as fertilizer. These results are close to those of Daud et al. [23]. On the other hand, these values are slightly higher than those of Vriesmann et al. [24] which had 85.9% for dry matter and 87.7% for organic matter. In addition, Ba et al. [25] had high values, i.e., a dry matter content of 91.5% and 93.3% for organic matter. As regards the lignocellulosic composition, there are 40.1% crude fibers, 35.3% lignin and 41.4% cellulose. These results are in line with those of Daud et al. [23] who for these same compounds had 42.22%, 38% and 43.13% respectively. In addition, these contents are higher than that of Ba et al. [25] whose work revealed a crude fiber content of 36.6% against 21.4% of lignin. Such lignin (between 10 and 35%) and cellulose (between 20 and 50%) contents immediately make cocoa shells a lignocellulosic biomass according to Balakrishnan et al. [26]. All these results demonstrate the variability in the composition of cocoa shells. This can be explained by differences in the soil composition of the place of origin, the difference in the degree of maturity as well as the harvest period. But the fact remains that cocoa powder constitute a promising source of loaders that can be used as reinforcement. The results are given in Table 3 below.

Table 3 Chemical composition of cocoa powder hulls

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

3.1.2 Thermal Analysis

The ATG-DTG curves of the cocoa hulls powder are shown in Fig. 1. Thermogravimetric analysis (ATG) (in red) provides the loss of mass of the material during thermal degradation. The heat treatment phenomenon is described by Verma et al. [27]. The second derivative of thermogravimetric analysis (DTG) (in blue) gives the degradation temperatures of the hulls as defined by Balakrishnan et al. [26].

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

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Observation of the curve shows that around 440 °C, the biomass has been completely consumed. Fig. 1 shows four main phases of mass loss. The first mass loss of 8.00% by weight is located between 40 to 100 °C and has a peak around 86 °C; it corresponds to the evaporation of the water present in the hulls. The second loss of mass correspond to mass loss of 64.50% is observed between 300 and 400 °C, with a DTG maximum at 321 °C. It is assigned to the degradation of polysaccharides (hemicelluloses and cellulose) in the fiber of cocoa hulls [27]. The third loss of 22, 60% observed between 321 and 401 °C would be due to the degradation of cellulose; in fact, cellulose would degrade around 401 °C. Between 401 °C and 423 °C, a loss of 5.12% is observed having a peak around 423 °C, which would be due to the degradation of the lignin at 423 °C [27].

It was noted that these results are in agreement with the chemical composition of cocoa hulls: the major constituents of which are cellulose, hemicellulose and lignin; they degrade with a strong loss of mass [28]. In addition, hemicelluloses decompose at low temperatures because their molecular chains are short. In addition to their low molecular mass, they have fewer regular structures in their chains [28]. The curve of Fig. 1 shows four thermal phenomena for cocoa hulls. One endothermic peak observed at lower temperature around 86 °C and three exothermic peaks range between 321 °C, 401 °C and 423 °C. The first thermal incident was attributed to evaporation of water molecules from fibers, the second third and fourth respectively represent the degradation of holo-celluloses, celluloses and lignin from the coffee hulls [28]. Table 4 give the different types of thermal phenomena that occur during the thermal analysis of the sample.

Table 4 Types of thermal phenomena

	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

3.1.3 Fourier Transform Infrared Analysis

As shown in Fig. 4 and Table 5, the FTIR analysis of cocoa hulls powder, shows different peak intensities. Five main groups of adsorption bands emerge, namely those between 3000 - 3500 cm⁻¹, 3000 - 2500 cm⁻¹, 2000 - 1500 cm⁻¹, 1000 - 500 cm⁻¹. The wavenumber bands between 3000 cm⁻¹ and 3500 cm⁻¹ are 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 characteristic of CO elongation. The absorption band of 1000 cm⁻¹ corresponds to the CC bond.

Fig. 2 Infrared of raw cocoa shell powder

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

Number of waves (cm ⁻¹)	attribution
3315	√(OH) of lignin and polysaccharides
2927	√ _{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

3.2 Mechanical Characterization of Composites Materials

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

Table 6 Mechanical properties of composite

%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

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. Table 6 show that the tensile strength of the composite increases with the amount of load introduced 10% to 30% and is equivalent to a value of 55.8 to 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 allows good adhesion and contributes to the densification of the composite. Beyond 30% of the powder content, the resistance to decrease which 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

The 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 which 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.

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 addition of the powder helps to increase the water absorption rate as more lignocellulosic powders are added to the composite, which means that more hydrogen bonds have formed between the water molecules and the OH group in powders. The same 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 less surface interaction between the matrix and the powder when mixed, resulting in higher water absorption [31].

3.3 Chemical Stability

Table 7 shows that after ten 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.

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

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

4. Conclusion

The paper studied the chemical stability and mechanical properties of bathroom wall composites manufactured from recycle polyethylene terephthalate mixed with cocoa hulls powder. Organosolv treatment of cocoa powder hulls enhanced the quality of the cocoa powder hulls by removal of hemicellulose and lignin thereby increased 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 hulls powder blend has higher tensile, flexural and impact strength compared to other blends. Recycled PET/30% cocoa hulls powder blend was also found to have good properties in term 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.

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