Mechanical properties, visual aspect, microstructure and life cycle assessment approach (LCA) focused on PP/wood flour biocomposites

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ABSTRACT

Increasing attention has been more given since a few years to natural fibre reinforced composites, i.e. biocomposites, due to the renewability of the raw materials, their potential composting and their good specific mechanical properties in comparison to conventional glass fibre reinforced composites. However, the environmental impacts of their end-of-life (EoL) treatments need to be evaluated before products are produced and installed commercially. EoL options between incineration, landfill and recycling have to be compared. The biocomposites considered in this study are polypropylene (PP) reinforced with wood flour (PP/WP, 10wt% and 30 wt% WF, injection molded) bioccomposites commonly used as decking in building sector

First, mechanical properties after different combination of degrading conditions such as reprocessing (up to 7 cycles,) and natural weathering were evaluated to estimate the recycling potential of biocomposites. Results were related to changes in the visual aspect and microstructural analysis. Incineration option is considered through the total heat release values. Finally, LCA was carried out to compare three EoL treatments.

1 INTRODUCTION

The wood-plastic composites (WPCs) mainly concern thermoplastic polymers reinforced by wood fibres or flour and are widespread in outdoor decking applications. The most common polymer matrices are polypropylene (PP), polyethylene (PE) and polyvinyl chloride (PVC). With the growing use of WPC, their end-of-life issue is expected to become larger and increasingly difficult and expensive. Assessing WPC recycling capability is up to now a challenging economic and scientific goal.

Several previous studies investigated the reprocessing of WPC or natural fibre reinforced composites. Positive results were found as satisfactory properties and a good aptness to be recycled were observed [i]. Despite these favourable tendencies in results, thermal degradation was found to be the major issue in WPC reprocessing. Indeed, many studies showed that polymer matrices tend to degrade by chain scissions under severe thermal and stress cycles [ii] [iii]. Vegetal fibers or flours are also prone to degrade under these conditions: decreases in particle size and particle rigidity as well as chemical attrition were consensually reported [iv] [v].

A major degradation factor during the life cycle of WPCs is outdoor exposure which is mainly characterized by a combined UV/humidity exposure. Weathering performed on WPCs generally induced a mechanical property alteration, a surface erosion with flouring effect, a surface yellowing and bleaching, a surface cracking, crystallinity changes and a dimensional instability (swelling and shrinking) [vi].

Few studies deal with the reprocessing of weathered polymer and composite materials. Jansson et al. [vii] studied the reprocessing of oxidised post-consumer PP/PE copolymers. They found that the elongation drops considerably after each ageing step and returns approximately to the initial value after each extrusion cycle. Three combined mechanisms were proposed: i) changes in the crystallinity ratio, ii) degradation located at the surface and iii) dilution of degraded polymer chains after re-extrusion. Besides, a synergistic effect between extrusion and ageing was evidenced as the combination degrades stronger than reprocessing or ageing does separately. Luzuriaga et al..

Concerning the reprocessing of weathered PP based WPCs, our previous work [viii] highlighted the same trends than Jansson et al. with the recovering of mechanical properties after reprocessing of artificially UV weathered samples. Numerous technics were used to understand this “regeneration” phenomenon: SEC measurements, rheological and DMTA tests evidenced that the reprocessing allows the degraded chains and the non-degraded ones to be mixed together at the molten state to obtain a homogenized material. A PP matrix recombination also occurs as the chain length increases and the carbonyl concentration decreases after reprocessing.
To consolidate those conclusions about the reprocessing behaviour of weathered WPCs, this work aims to determine and understand the influence of a one-year natural UV weathering on the reprocessing of PP based WPCs. Injected neat PP and PP/wood flour (10%w and 30%w wood content) were submitted to a natural outdoor exposure followed by a reprocessing cycle (grinding and injection). The evolution in surface visual aspects was followed by optical microscopy. The mechanical behaviour was measured thanks to tensile and Charpy impact tests to understand the material physical degradation.

2 MATERIALS AND METHODS

2.1 Materials

Polypropylene (PP) used in this study is a standard homopolymer PP H733-07 grade supplied by Braskem Co. (Brazil) with a melt flow rate of 7.5g/10min (230°C, 2.16kg) according to ISO 1133 standard. Maleic anhydride grafted polypropylene (MAPP) with a 1%w/w grafting rate is used as coupling agent and provided by Arkema Co. (France) under the trademark Orevac® CA100. It was dry-mixed before processing at 3%w/w of the PP. The wood flour is based on spruce wood with a particle size included in the 200 µm to 500 µm range and is obtained from AFT Plasturgie Co. (France). The wood flour was added at 10%w/w and 30%w/w in the PP/MAPP matrix.

2.2 Compounding

Prior to extrusion, the polymer granules have been dried at least 4h at 80°C and the wood flour, 15h at 80°C. These drying conditions induce a decrease of WF moisture content from around 4.8% to 0.4% using a Karl Fisher method with a sample heated at 150°C.
In the subsequent phase, the PP matrix and the wood particles were mixed together in a BC21 Clextral co-rotating twin-screw extruder. Its L/D ratio is 36 with a 25 mm screw diameter and a 900 mm screw length. The heating barrel is composed of 12 modules. Polymer pellets were fed in module 1 and the fiber incorporation was made through a feeding hopper located on module 5. Temperature was set at 180°C along the barrel. The screw speed was arbitrarily fixed at 300 rpm, with a total feeding rate of 4 kg/h.
Extruded compound rods were cooled into water and rapidly dried by air pulsing before the granulating step. Pellets were kept overnight at 80°C in a vacuum oven beforehand to remove the residual humidity.

2.3 Injection molding and grinding cycles

The pellets were injection molded on a Krauss Maffei KM50-T180CX. The temperature was fixed at 210°C along the barrel. The mold was kept at 25°C by a water cooling system. The plastification and injection speeds were set respectively at 120 rpm and 60 $cm^3.s^{-1}$. The samples were injected to obtain dog-bone samples ISO 1A according to ISO 527-2. Some of them were grinded and injected multiple times while a set was characterized.
The grinding process was performed in a RETSCH SM300 cutter mill to obtain flakes. The grinding process was carried out at 700 rpm at room temperature with an 8 mm sieve. The flakes were stored at room temperature and vacuum dried overnight at 80°C before injection molding. This protocol was

accomplished 1, 3, 5 and 7 times in order to purvey samples called P1, P3, P5 and P7 according to the number of passes they underwent.

2.4 Wood flour characterizations

2.4.1 Scanning electron microscopy (SEM)

Wood flour dispersion and cell wall were analyzed through scanning electron microscopy (SEM). An Environmental Scanning Electron Microscope (ESEM) Quanta FEG 200 was used.
For the wood flour analysis within the composites, cross-section samples of composites were prepared through the same methodology. They were obtained from the middle part of the dog-bone samples. Then, they were embedded in epoxy resin and the surfaces were carefully polished.
The magnifications were x80 to determine the dispersion, x2 000 to assess the general morphology of the wood cells and x10 000 in order to observe the wood/matrix interface.

2.5 Processes and natural weathering

The processing methodology is summarized in Figure 1 and the successive stages are detailed below.
Beforehand, the wood flour is dried 15h at 80°C to remove moisture from the particles. Then, the PP matrix and the wood particles are mixed together in a BC21 Clextral (France) co-rotating twin-screw extruder (step 1). Its L/d ratio is 36 with a 25 mm screw diameter and a 900 mm screw length. Temperature is set at 180°C along the barrel. The screw speed is fixed at 300 rpm with a total feeding rate of 4 kg/h. The extruder is equipped with a 4 mm die diameter. The extruded compound rods are cooled into water and granulated. Pellets are kept 6h at 80°C (step 2) in an air-pulsing apparatus.
The pellets are injection moulded to produce normalized samples on a Krauss-Maffei KM50-T180CX. The temperature is fixed at 210°C along the barrel. The mould is kept at 25°C by a water cooling system. The plasticization and injection speed are set respectively at 120 rpm and 60 $cm^3.s^{-1}$. The samples are injected to obtain dog-bone samples 1A according to ISO 527-2 (step 3). Samples are characterized at this step to evaluate their initial stage (INIT).

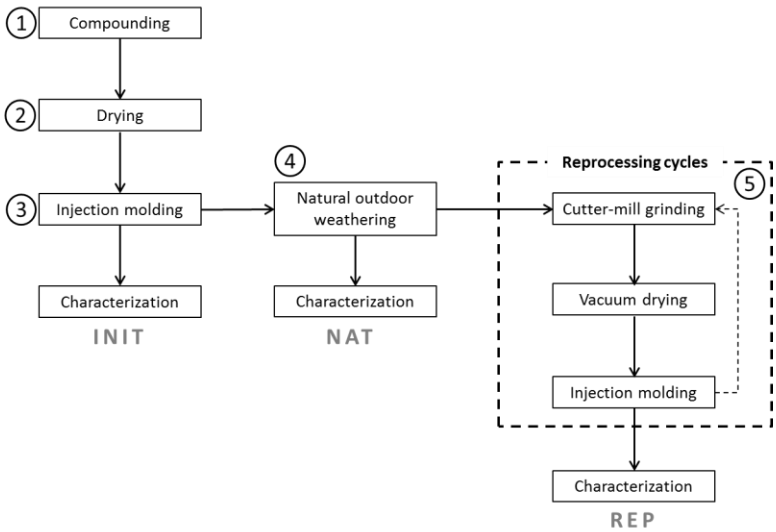


Figure 1 - Stages of the weathering and reprocessing stages

ISO 1A samples are exposed according to EN ISO 877 standard in natural site in the South of France on galvanized steel racks (step 4). The exposure included climatic conditions from the four annual seasons. According to the mentioned standard, the samples are fixed on the racks at an angle of 45° with the ground on a south facing flat land.

Aged samples are characterized (NAT stage), then grinded and injected to simulate recycling (step 5). The grinding process is performed in a RETSCH SM300 cutter mill to obtain flakes. The grinding process is carried out at 700 rpm at room temperature with an 8 mm sieve. The flakes are stored at room temperature and vacuum dried overnight at 80°C before injection molding (REP stage).
Samples used in this study are detailed in Table 1.

Table 1 - Designation of the studied materials

Wood flour content by weight	Unaged INITial state	NATural UV aged	REProcessed after natural UV ageing
0%	PP_{INIT}	PP_{NAT}	PP_{REP}
10%	$PP/WF10_{INIT}$	$PP/WF10_{NAT}$	$PP/WF10_{REP}$
30%	$PP/WF30_{INIT}$	$PP/WF30_{NAT}$	$PP/WF30_{REP}$

2.6 Characterization of surface aspect evolution

Colour and texture changes of the sample surface are visually assessed with a Leica WILD M10 optical microscope. Pictures are taken at x10 and x50 magnifications with a Leica DFC 420 camera. Pictures from a same material are taken with the same light intensity to properly discern colour changes.

2.7 Composite mechanical characterization

2.7.1 Tensile tests

Classical tensile tests were performed to determine mechanical properties of PP and PP/WF composites. The apparatus used for these tensile tests is a Zwick Z010 with a 10 kN load cell and a Clip-On extensometer for displacement measurements during modulus tests. These tests were performed according to the ISO 527 standard: the crosshead speed is set at 1 mm/min for the tensile modulus measurements, 50 mm/min for the break property measurements. Thus, Young modulus, strength and deformation were determined as a function of the reprocessing steps (P1, P3, P5 and P7). 5 measurements were carried out for modulus measurements and for strength and elongation at break measurements.

2.7.2 Charpy tests

Impact strength is measured with a Charpy pendulum impact tester ZWICK 5102. The tests are performed according to the ISO 179 standard and repeated for ten unnotched samples. A 4 J pendulum is chosen. The unnotched samples are sawn from the ISO 1A injected samples with 80 x 10 x 4 mm^3 dimensions.

3 RESULTS

3.1 Effect of reprocessing on biocomposites performance

3.1.1 Effect of the wood flour size on the composites properties during the processing cycles

3.1.1.1 Evolution of wood flour particles

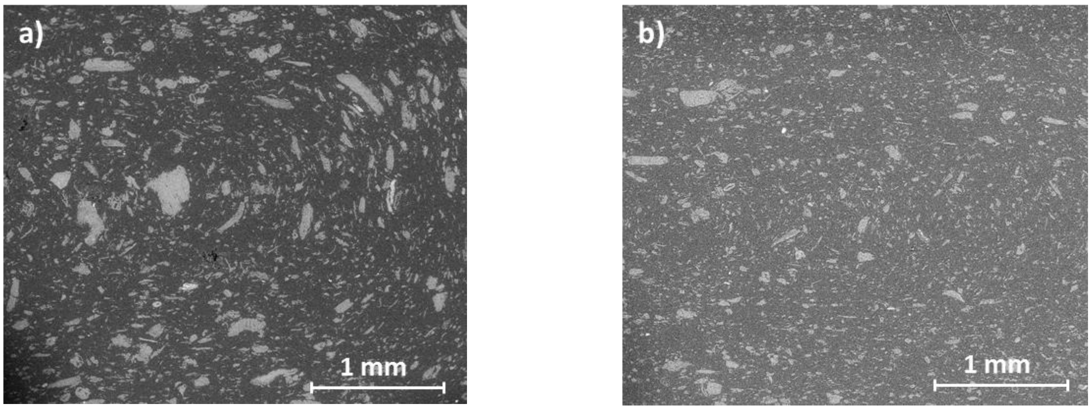


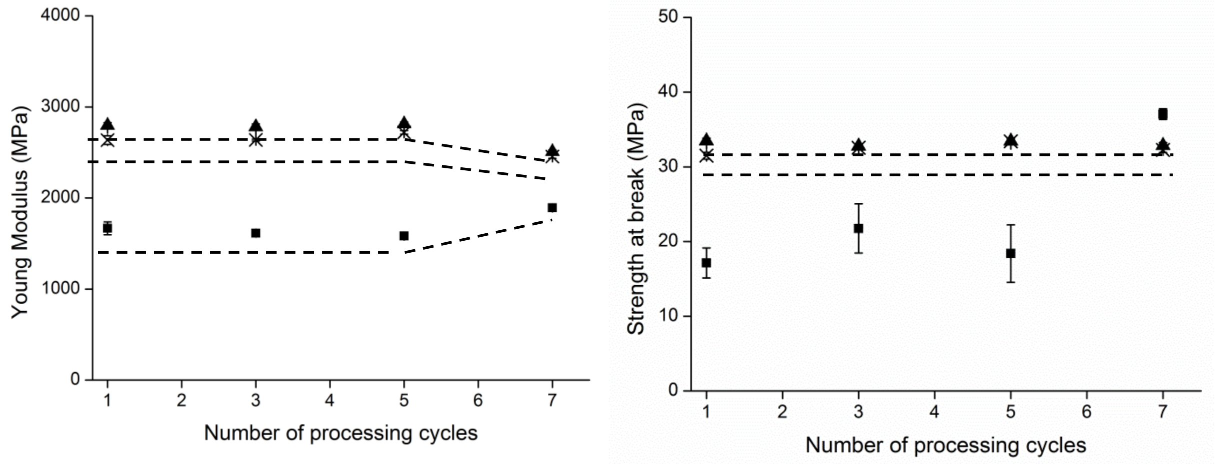
Figure 2: SEM micrographs of transversal cross sections of PP/WF at P1 (a) and at P7 (b) (x80)

0 presents SEM cross sections of PP/WF along the processing cycles. The wood particle morphology and size reveal to be modified after 7 cycles. A breakdown of the coarser wood particles can be observed due to high shear rates during processing and grinding steps. This tendency is the most visible where the numerous bundles are breaking progressively. It seems to be less sensitive thanks to its smaller initial size. As these coarse particles progressively disappear, the dispersion becomes more homogeneous along the cycles.

3.1.1.2 Evolution of composite mechanical properties

0 presents the tensile properties obtained for the neat PP and for PP/WF composites from 1 to 7 processing cycles. Concerning the behavior of neat PP up to 5 processing cycles, a slight increase in Young modulus and strength at break and a gradual decrease of elongation at break are observed. This suggests a prevalent chain scission phenomenon up to 5 cycles. At 7 cycles, an increase in modulus and strength at break occurs and suggests a possible simultaneous crosslinking mechanism.

a) b)



c)

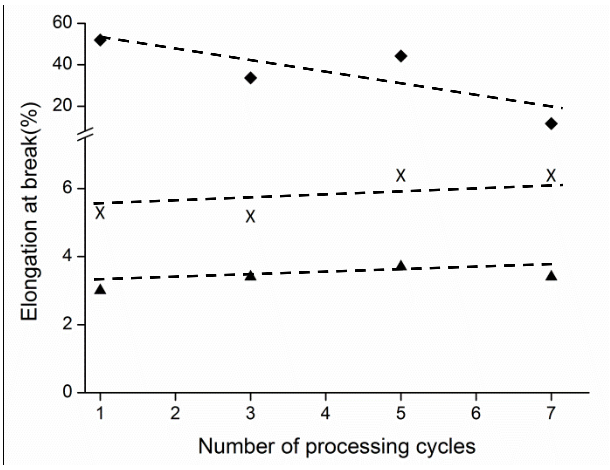


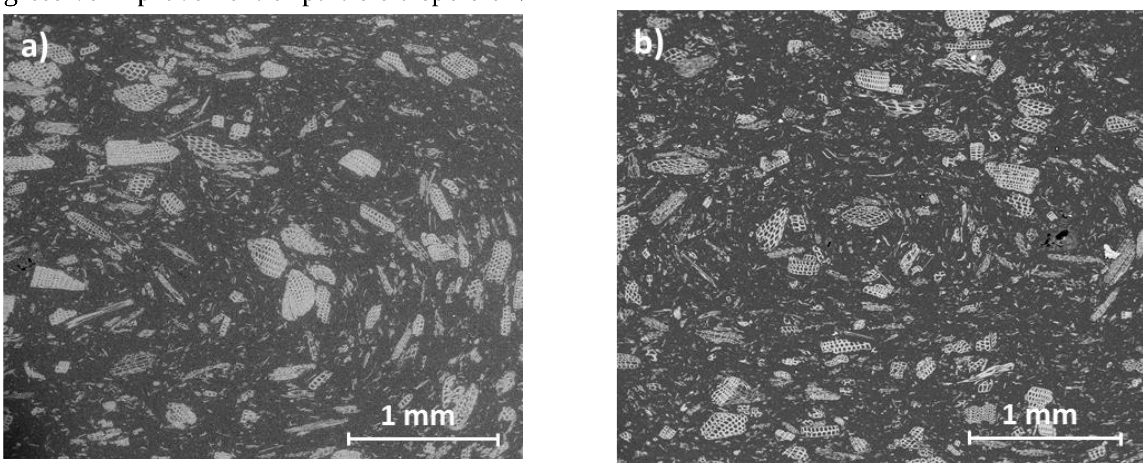
Figure 3: Young modulus (a), strength at break (b) and elongation at break (c) measured by *tensile test at 1, 3, 5 and 7 processing cycles of neat PP (■), PP/WF G1 (X) and PP/WF G2(▲)*

For every composite (G1 and G2), it can be noticed that the tensile stiffness is well preserved along 5 processing cycles and then decreases. The strength at break remains similar until the 7 processing cycles as a gradual increase of the elongation at break occurs. The good preservation of the tensile stiffness and strength along the processing cycles corroborate well the tendency found by Bourmaud and Baley [iv] for a flax fiber composite which reveals that the natural fiber bundles split into finer fibers during the process and that it is not detrimental to the composite mechanical performances. Otherwise, it is well known that higher fiber size produces higher strength and elasticity but lower elongation [xi] [xii] due to a favored crack propagation. It can be slightly observed on the present tensile results. Both composites show a slight increasing elongation with the processing cycles because the particle size lessens as the crack propagation.

3.1.2 Effect of the addition of PPgma on the composites properties during the processing cycles

3.1.2.1 Evolution of wood flour particles

By comparing SEM pictures of PP/WF and PP/PPgma/WF at P1 (**Erreur ! Source du renvoi introuvable.**), the wood particles show a similar breakdown because of the processing cycles with a progressive improvement of particle dispersion.



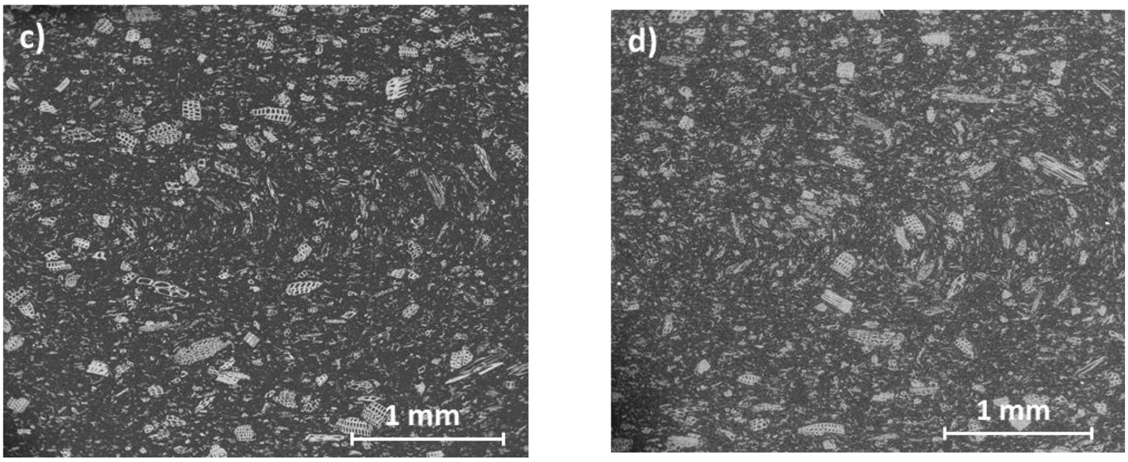


Figure 4: SEM micrographs of transversal cross sections of PP/PPgma/WF at P1 (a), P3 (b), P5 (c) and P7 (d) (magnification x80)

3.1.2.2 Evolution of composite mechanical properties

0 shows the tensile properties of the PP/PPgma/WF composites. It can be observed that the addition of PPgma induces greater stiffness and strength thanks to a compatibilisation effect as already seen by Kord [xiv]. Concerning the reprocessing behavior, the tendency is the same with the free-PPgma composite: the modulus and strength remain quite stable while the elongation increases because of the particle size reduction.

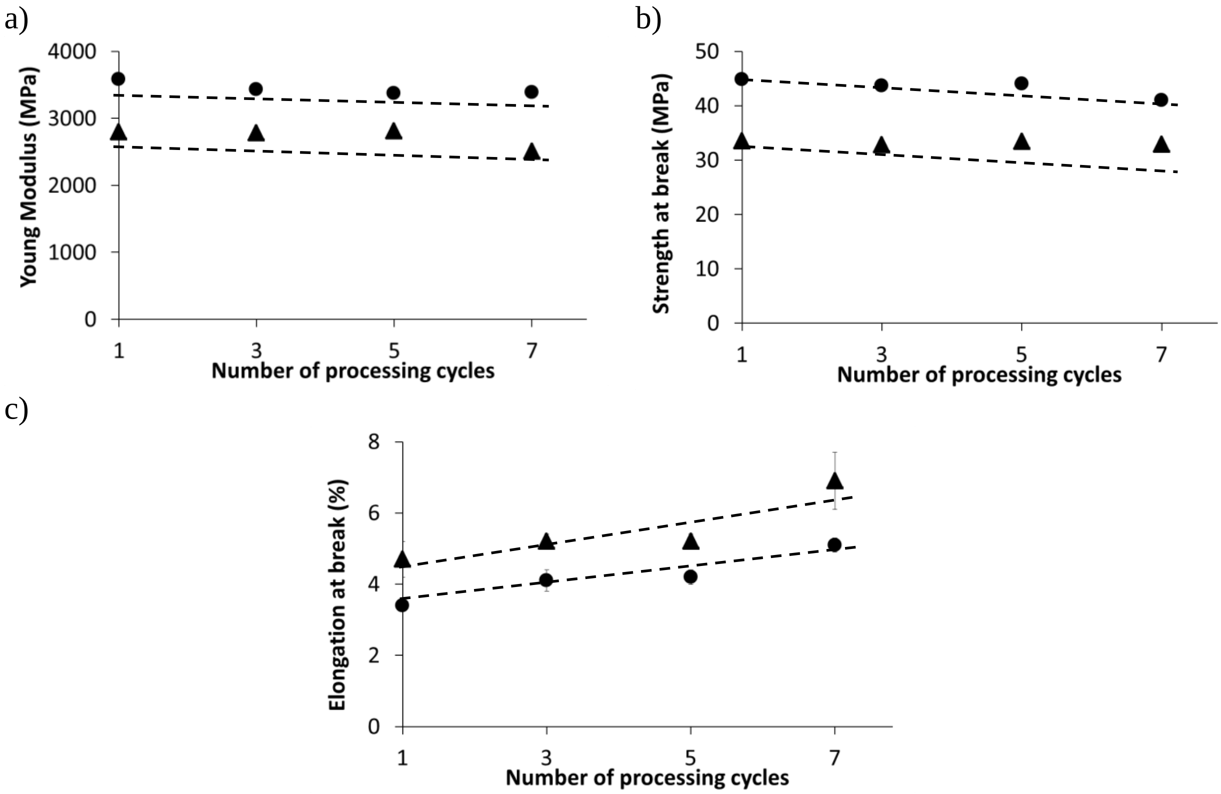


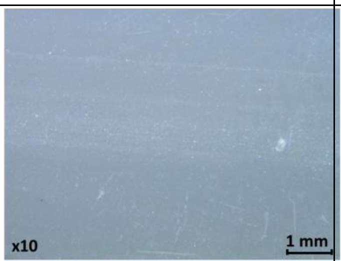
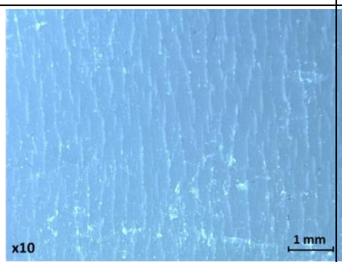
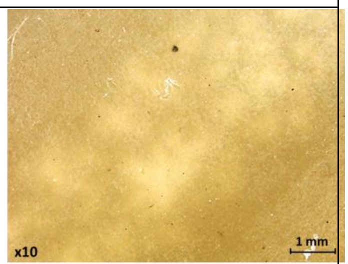
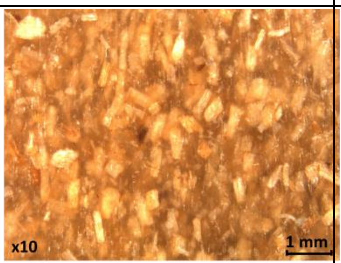
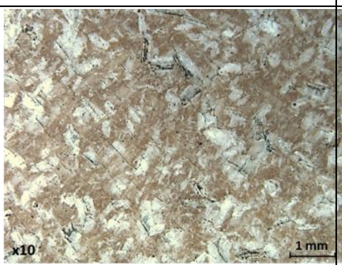
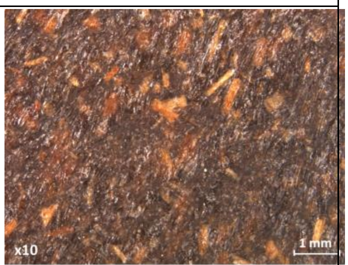
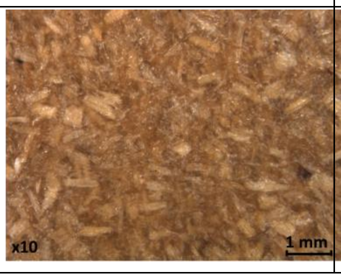
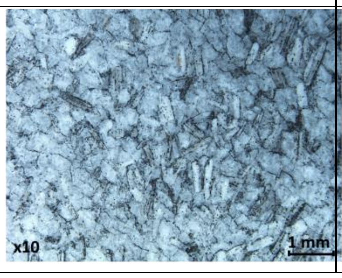
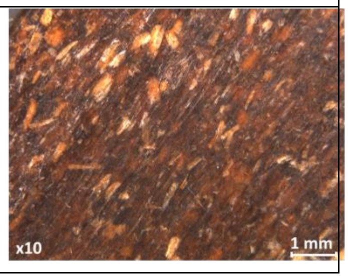
Figure 5: Young modulus (a), strength at break (b) and elongation at break (c) measured by tensile test at 1, 3, 5 and 7 processing cycles of PP/WF (▲) and PP/PPgma/WF (●)

3.2 Effect of weathering on the recycling potential of biocomposites

3.2.1 Evolution of the surface aspect

The sample surface aspect is presented in Table 2 for PP, PP/WF10 and PP/WF30 respectively as a function of the different stages (INIT, NAT and REP). Neat PP surfaces exhibit more important cracking phenomena due to photodegradation during outdoor weathering (NAT step) than during artificial UV weathering [xiii]. Moreover, the reprocessing step leads to a material displaying a strong yellowing effect. For both PP/WF10 and PP/WF30, outdoor weathering induces a wood particle bleaching and a protrusion of these particles at the surface with many cracks into the PP matrix as for artificial weathering. This degradation is so important that a flouring phenomenon is obtained on the exposed surface. The bleaching leads to an entirely white surface with no brown parts visible. The surface is whiter than in the case of artificial weathering [xiii]. It was previously shown that this wood bleaching is well-known and is mainly due to the lignin chromophoric groups absorbing in the UV/visible region. This photo-degradation is explained by two competing reactions in the literature [ix] [x]: the first one is lignin oxidation, which leads to the formation of paraquinone chromophoric structures, and the second one involves reduction of the paraquinone structures to hydroquinones, which leads to photo-bleaching. Moreover, some black particles are visible as aggregates accumulated into the cracks. These black aggregates are supposed to be airborne pollutants such as combustion residues from industrial or domestic activities or emissions from road transport in the surrounding area of the natural ageing site.
Then, after reprocessing, the surfaces recover a glossy aspect thanks to injection moulding process with no protrusion of the wood particles. The bleached aspect has disappeared but the global colour is much darker than the initial state. This is probably induced to a combined effect of the mixing and dilution of bleached parts and the darkening due to wood degradation during process.

Table 2 - Micrographs of sample surface for neat PP and PP/WF composites at the different stages: INIT, NAT and REP

	INIT	NAT	REP
Neat PP			
PP/WF10			
PP/WF30			

3.2.2 Evolution of mechanical behaviour

3.2.2.1 Tensile property variations

Figure shows tensile ultimate strength values for all studied materials at each stage (INIT, NAT, REP). A drastic decrease in the yield strength with the natural weathering was observed with loss of 60%, 18% and 14% for PP, PP/WF10 and PP/WF30 respectively. This can be ascribed to a chain scission mechanism by photodegradation. Then, the reprocessing step after natural weathering brings strength values up. PP, PP/WF10 and PP/WF30 recover 82%, 97% and 100% respectively of their initial strength. If comparing with artificial weathering, previous results showed no significant degradation of yield strength for similar materials. Thus, the natural exposure is more detrimental than the artificial ones under these conditions.

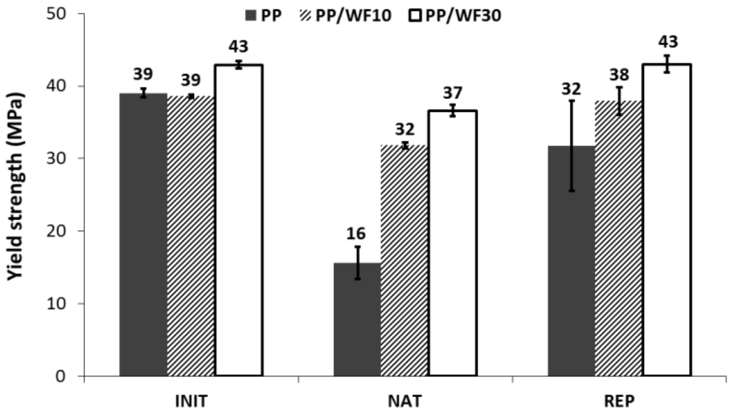


Figure 6: Tensile yield strength of PP and PP/WF composites at the initial state (INIT), at the weathered state (NAT), at the reprocessed state after weathering (REP)

Concerning the tensile elongation at yield (Figure), the addition of wood flour globally decreases the deformation rate because the PP chain mobility is restrained by wood particles. After weathering, neat PP exhibits a large drop from 7% ±0.3% to 1% ±0.2% due to chain scission but recovers little ductility after reprocessing (4% ±1.7%). Concerning PP/WF10, the weathering decreases the elongation from 4% to 3% but reprocessing does not have an impact on this elongation, remaining at 3%. PP/WF30 has an elongation of 2% for every stage and is not impacted by weathering and reprocessing in its elongation. PP/WF materials are the most stable against weathering and reprocessing as their evolution is very low compared to neat PP. The addition of wood flour seems to stabilize the PP polymer against degradation. This observation is in accordance with several previous studies that proves the antioxidant role of lignin. Thus, high wood content leads to high amount of lignin, hindering mechanical degradation. In the case of artificial weathering, neat PP is less degraded (4% ±0.1%) and recovers better its value of 8% ±0.1% in elongation after reprocessing. The PP/WF composite elongations stay in the same range and are not impacted by the artificial weathering. The similar tendency of tensile property recovery after reprocessing was observed only for artificially weathered PP samples, PP/WF composites being lowly impacted by weathering and reprocessing.

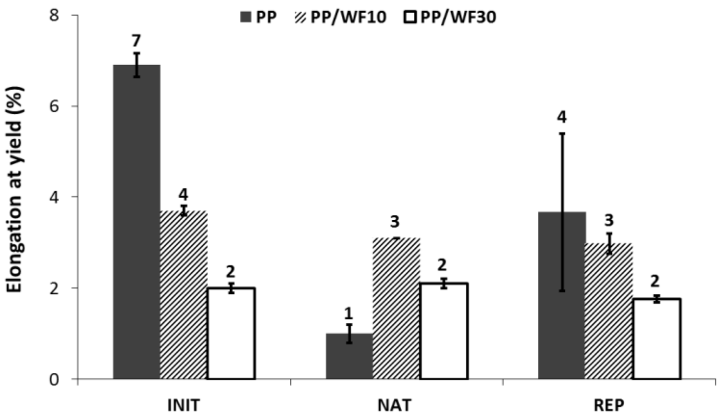


Figure 7: Tensile elongation at yield of PP and PP/WF composites at the initial state (INIT), at the weathered state (NAT), at the reprocessed state after weathering (REP)

3.2.2.2 Impact strength variations

Results from un-notched Charpy tests are plotted in Figure . No break for neat PP is observed at the initial state thanks to its ductile property. PP becomes brittle after the natural weathering step with very low impact strength (1± 0.1 kJ/m²) but return to its ductile state (25±3.9 kJ/m²) after reprocessing. This high impact strength value confirms the tendency found with tensile elongation after reprocessing. Concerning PP/WF10 and PP/WF30, the same tendency is observed with a drop after weathering, and "regeneration" of impact properties after reprocessing. This phenomenon is less significant for PP/WF30 probably due to the known stabilizing effect of lignin.

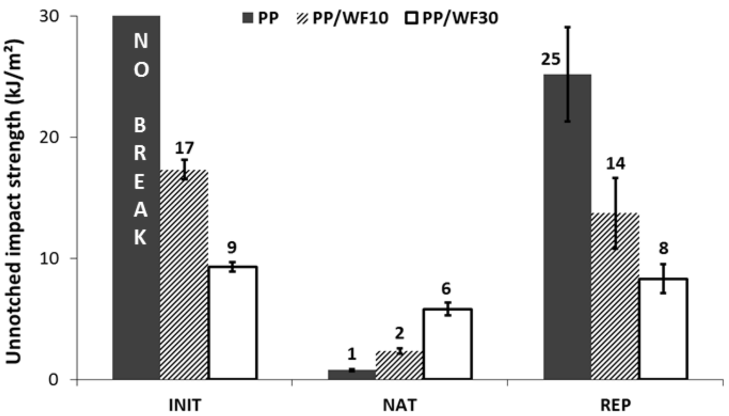


Figure 8: Un-notched impact strength by Charpy tests at the initial state (INIT), at the weathered state (NAT), at the reprocessed state after weathering (REP)

To sum up the results obtained from static mechanical tests, the addition of wood flour reduces the ductility of PP and reduces the sensitivity against degradation by natural weathering and reprocessing thanks to the antioxidant role of lignin. A "regeneration" tendency can be observed with the reprocessing as it enables to recover a part of tensile ultimate strength, elongation at break and impact strength. The same tendencies were previously obtained for artificial weathered and reprocessed samples. A competition between chain scission and chain recombination mechanisms and a transfer of degraded chains from amorphous phases to crystalline phases are suggested. To complement these hypotheses, the polymer microstructure is investigated further on.

3.3 Environmental impacts of EoL treatments of biocomposites

LCA was applied to the treatment of PP/WF wastes in order to predict the best environmental practices for EoL management. Recycling EoL scenario was presented the lowest environmental impacts (climate change, freshwater eutrophication, terrestrial acidification, photochemical oxidant formation, ozone depletion, fossil depletion, human toxicity, freshwater ecotoxicity) followed by incineration and then landfill.

3.3.1. Climate change

Climate change potential impacts of end-of-life options for the biocomposite studied are presented on Figure . For both composites, recycling presents the lowest net total impact score, followed by landfill and incineration. Transport step is negligible compared to other life cycle steps. The process of incineration is responsible of important emissions of CO_2, which are not compensated by the credit obtained from the production of electricity. As composites degradations in landfill are limited, emissions of this process are quite low and only due to the facilities uses. Recycling credit is important, indeed, the production of any polymer is at the origin of major emissions of CO_2, mainly coming from all the energy consumed during the entire process (fuel extraction, cracking, polymerization, etc...

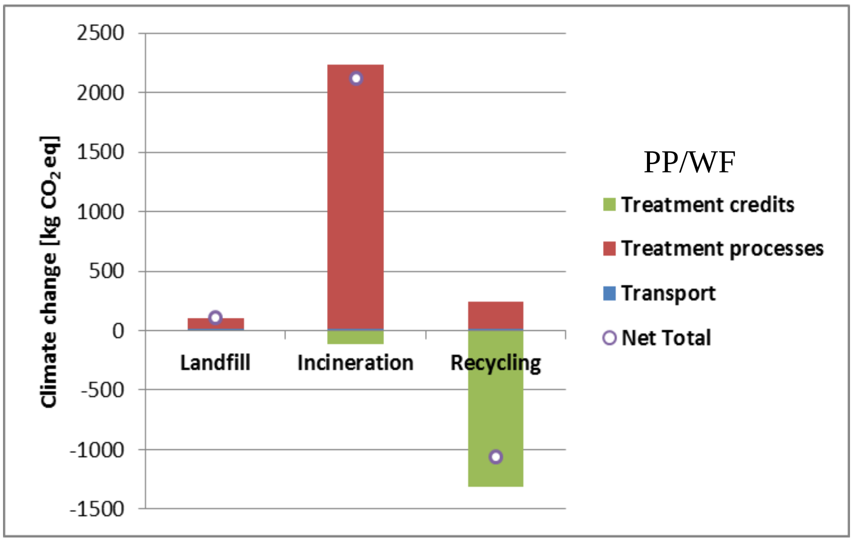


Figure 9 : Climate change impact scores for the different end-of-life options of PP/WF composite

3.3.2. Human toxicity

Human toxicity potential impacts are presented on Figure . Recycling options present the lowest net total scores, followed by incineration and landfill. Landfill processes present the highest impacts for the biocomposite. Indeed, during landfill, leachates bring residual heavy metals from the catalyst of the polymer to groundwater. Incineration processes also has important human toxicity impacts, due to toxic emissions in air. Credits from the recycling of biocomposites enable to compensate impacts of the recycling processes.

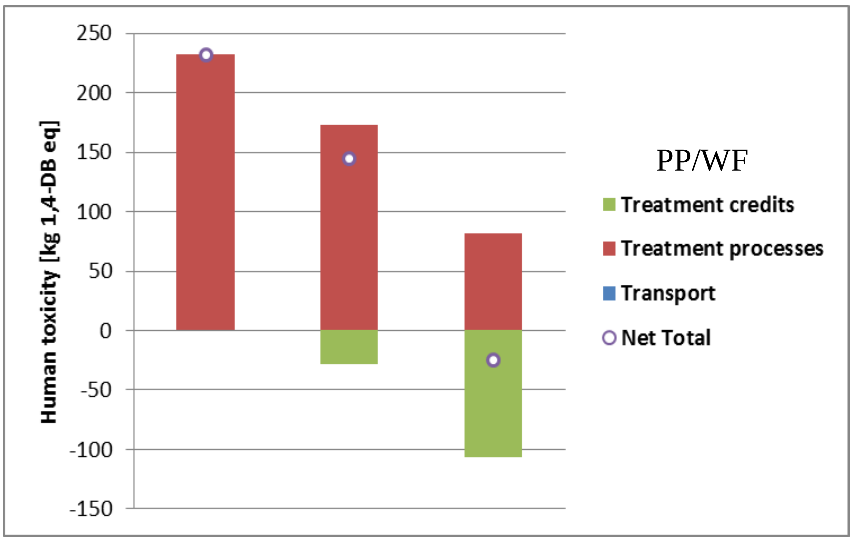


Figure 10 : Human toxicity potential impact scores for the different end-of-life options of PP/WF composite

3.3.3. Freshwater ecotoxicity

Figure show the freshwater ecotoxity potential impacts for PP/WF composite. As for human toxicity, landfill options present the highest net scores, followed by incineration and recycling. Leachates of landfill sites are also at the origin of a pollution of water. For PP/WF, avoided impacts coming from production of electricity and recycling are negligible compared to the processes impacts.

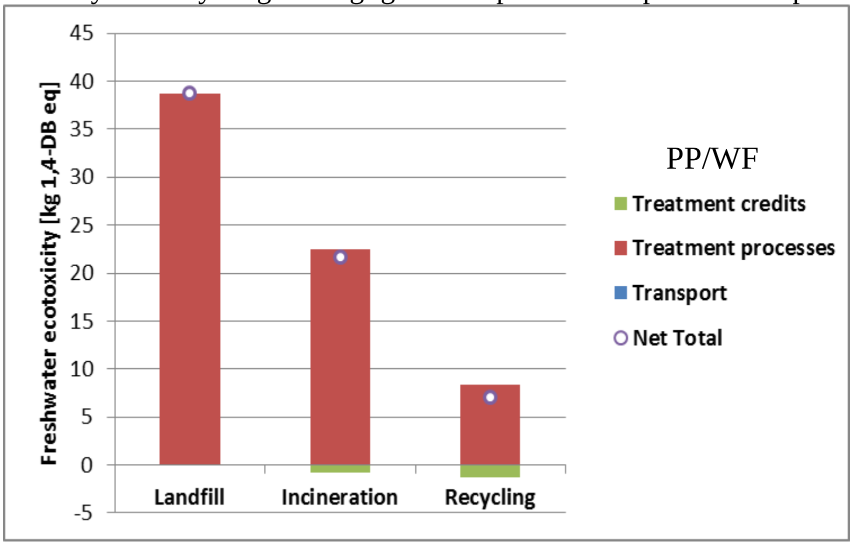


Figure 11 : Freshwater ecotoxicity potential impact scores for the different end-of-life options of PP/WF composite

Recycling option presents the lowest environmental impacts for both composites, followed by incineration with energy recovery and landfill. In most cases, benefits from the production of recycled materials or energy compensate the impacts of waste treatment processes. Thus, the presence of natural fibres in PP/WF composite does not modify the waste hierarchy obtained for PP alone.
Recycling option needs a minimal flow of wastes to be economically viable and also an existing market for the recycled composite.

4 CONCLUSIONS

The main conclusions of this study are the followings:

i) correlations may exist between non-destructive parameters determined through a visual analysis and mechanical characteristics,
ii) there is a beneficial effect of reprocessing thanks to a mechanical property recovery after weathering (artificial or natural)
iii) recycling end-of-life scenario is the option that induces the lowest environmental impact

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