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A circular economy use of recovered sludge cellulose in wood plastic composite production: Recycling and eco-efficiency assessment

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ABSTRACT

This paper presents a novel development of sludge cellulose plastic composite (SPC) in line with the circular economy concept by using recovered sludge cellulose from wastewater treatment plant (WWTP). Bearing the aim of replacing the wood in wood plastic composite (WPC) with sludge cellulose, WPC was developed in parallel for determining the substitution potentials. In order to maximise the integration of properties, maleic anhydride (MA) and vinyltrimethoxysilane (VTMS) coupling agents were employed to refine the interfacial bonding of both SPC and WPC. In line with the main aim of circular economy – to decouple the economic value from the environmental impact, eco-efficiency analysis was performed for the developed process. The results showed that the tensile and flexural strength of the composites were substantially enhanced after both treatments, while MA appeared to be more efficient than VTMS in the refinery of interfacial bonding. Scanning electron microscope (SEM) analysis confirmed the improvement of interface by identifying well embedded and firmly bonded wood flour or sludge cellulose in the matrix. WPC was marginally more thermally stable than SPC, while SPC suggested comparable flexural properties. Eco-efficiency assessment results showed that the SPC had better environmental and economic performance than the WPC. The latter turns sludge cellulose as a promising sustainable alternative to wood or natural fibres in the production of WPC.

1. Introduction

Wood plastic composite (WPC) has grown rapidly in recent years mainly due to its increasing applications in building and construction products, automotive components, and industrial and consumer goods (Keskisaari and Kärki, 2018; Zhou et al., 2016; Kazemi Najafi, 2013). Wood components in WPC are generally used in sawdust form or small fibres, and typically comprise between 30 and 70 percent of the final product. The European Bioeconomy Strategy towards a sustainable growth promotes the use of wood and wood based products (Sommerhuber et al., 2015). Wood resources are facing a strong competition between various utilisation industries, i.e. energy, biofuel, chemicals, and materials (Bais-Moleman et al., 2018; Vega et al., 2019). It has been reported that there will be no sufficient wood from sustainably managed forests for the competitive markets of material and fuel in 2030 (Sommerhuber et al., 2017). The concern of resource scarcity and product profitability has driven the wood-based products sector to efficiently recover and reuse lignocellulosic by-products and wastes to close economic and ecological loops of resource flows.

Recently, various types of wood wastes (e.g. sawdust, off-cuts and shavings) and non-wood wastes (e.g. sugarcane residues, jute fibre, coconut husk, cotton stalk, rice husk and other lignocellulosic wastes) have been applied to produce WPC, aiming at maximising the economic and environmental benefits (Félix et al., 2013; Mitchell et al., 2014). In terms of the property and performance of the resulted WPC, wood and other lignocellulosic wastes had demonstrated comparable results to their virgin counterparts (Souza et al., 2018). Similarly, the compatibility and interfacial bonding between the filler and plastic matrix are the indispensable issues to be addressed when the waste resources are employed in the manufacture of WPC, in order to achieve adequate integration of properties. With respect to the commercial production of WPC, incorporating coupling agents is probably the best available and feasible strategy for its interface optimisation among numerous physical and chemical treatment methods (Zhou et al., 2017).

Sewage sludge has commonly been thought of as waste, and a problem to dispose of Gorazda et al. (2018). Wastewater treatment plants (WWTP) have recently moved from the concept of “waste treatment”, to the concept of “water resource recovery facility”

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(WRRF). This transformation from pollutants removal to value added resources (water, energy and materials) is the core of the transition to a circular economy (Bilitewski, 2012). It is important to apply innovative processes to recover energy-efficient materials from wastewater. As part of the EU funded SMART-Plant H2020 project (http://smart-plant.eu/), the pilot system operated at Geestmerambacht WWTP (CirTec, Netherlands) intends to prove that sludge can be a valuable product from which resources can be recovered and reused. This is the first full-scale installation worldwide that uses sewage to produce a significant amount of high-grade cellulose (150–300 kg/day), which can be reused in commercial products (e.g. Asphalt) (http://www.cirtec.nl/en/portfolio/cellvation/). Specifically, a Salsnes Filter system has been installed at Geestmerambacht for primary treatment, separating cellulose fibres from toilet paper in the wastewater to produce a highly concentrated sludge. The sludge is then sent for post-processing inside the treatment plant. The end product is marketable cellulose that has been cleaned, dried and disinfected.

It is deemed that the recovered cellulose fibres from WWTP can be used to produce paper products, building and construction materials (e.g. asphalt) as well as bioplastics (Ruiken et al., 2013; van der Hoek et al., 2016). In the last years, the recovery of sludge cellulose has received significant attention, unfortunately its further utilisation was very rarely reported. In the present work, sludge cellulose (SC) recovered from Geestmerambacht WWTP (CirTec, The Netherlands) is uniquely used as a raw material to replace the wood flour (WF) in WPC, such to develop sludge cellulose plastic composites (SPC). The novel application of sludge cellulose in WPC production on one hand points to an innovative way to achieve valorisation of the recovered resource from WWTP, on the other hand provides a promising solution to improve the sustainability of WPC industry by reducing its environmental impact and manufacturing cost and alleviating resource competition with other industrial sectors. The focus of the work was to formulate compatible SPC by the use of maleated and silane coupling agents, and thus to determine its substitution potentials in terms of material properties, manufacturing cost and environmental impacts by carrying out mechanical and thermal property analysis, life cycle impact, cost assessment and eco-efficiency analyses.

2. Materials and methods

2.1. Materials

The materials and additives used in the work were summarised in Table S1. All the raw materials and additives were stored in a cool dry place before uses.

2.2. Formulation of composites

The formulation of untreated and treated WPC and SPC with specific ratios is summarised in Table 1. All the composites were processed as follows: the required amount of HDPE for each batch was first placed in a Brabender Plastograph twin-screw mixer (with Cam blades for mixer type N50EHT) and allowed to completely melt at 50 rpm and 180 °C for 2 min, and subsequently mixed with lignocellulosic filler (wood flour or sludge cellulose) for 3 min to obtain uniform mixture. The lubricants, initiator and coupling agents were thus added into the system and mixed for another 10 min to allow their reaction with the raw materials. The lubricant was used at 2.5%, and the combined dosage for initiator and coupling agent was 3%. These compositions were selected based on our previous work (Zhou et al., 2017) and the dosage used in the commercial production of WPC. The resulted mixture was thus ground to pellets by using a Retsch cutting mill (SM 100, Germany). The ground blends were compression moulded on an electrically heated hydraulic press. The dimension of the mould was 20 cm (L) × 20 cm (W). Hot-press procedures involved 10 min preheating at 180 °C with no load applied followed by 5 min compressing at the same temperature under the pressure of 9.81 MPa, and subsequently air cooling under load until the mould reached 40 °C.

2.3. Mechanical property analysis

Tensile and flexural properties of the composites were determined at a speed of 1 mm/min according to the standard BS EN ISO 14125 and BS EN ISO 527-2:2012 respectively, on an Instron 5900 testing machine with 30 kN load capacity. Three point bending was used for measuring the flexural properties. The dimension of flexural specimen was 80 mm (L) × 10 mm (W) × 4 (T). The ratio of span length to specimen thickness is 16. The specimen for tensile tests was dumb-bell-shaped type 1A (BS EN ISO 527-2:2012). For each sample, the tensile and flexural property is the average of six measurements.

2.4. Thermal property analysis

Thermogravimetry (TG) curves were determined with a thermal analyser (Netzsch STA 449 F3, Germany) in a nitrogen atmosphere. The weight of samples tested was between 5 and 10 mg, and the measurements were carried out from room temperature to 600 °C at a heating rate of 10 °C/min.

2.5. Microstructure analysis

The SEM observation of the fracture surface (cross section) of the composites after tensile test was conducted on Zeiss Supra 35VP SEM operating at 20 kV with secondary and backscatter electron modes. All the samples were conductively plated with gold by sputtering for 45 s before imaging.

2.6. Eco-efficiency assessment

In line with the main aim of circular economy – to decouple the economic value from the environmental impact, eco-efficiency analysis was performed for both WPC and SPC products

Table 1
Formulation of the composites.

Sample	HDPE (%)	Cellulosic filler		Lubricant and compatibiliser		Initiator	Coupling agent	
		WF (%)	SC (%)	TPW 709 (%)	12HSA (%)	Peroxide (%)	MA (%)	VTMS (%)
Untreated WPC	55	40	0	2.5	2.5	0	0	0
Untreated SPC	55	0	40	2.5	2.5	0	0	0
MA treated WPC	52	40	0	2.5	2.5	0.3	2.7	0
MA treated SPC	52	0	40	2.5	2.5	0.3	2.7	0
VTMS treated WPC	52	40	0	2.5	2.5	0.3	0	2.7
VTMS treated SPC	52	0	40	2.5	2.5	0.3	0	2.7

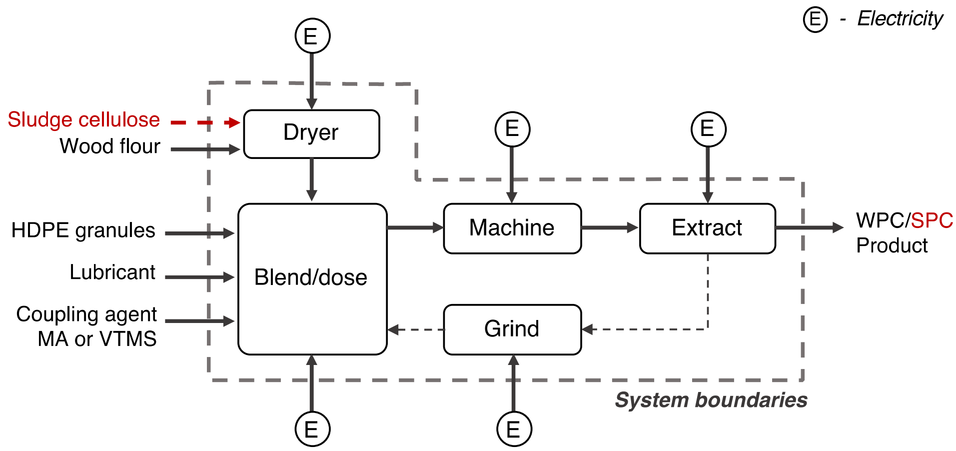

Fig. 1. System boundary and life cycle flow diagram.

(Tunn et al., 2019; Domenech and Bahn-Walkowiak, 2019). Eco-efficiency relates the environmental performance of a product system to its product value or costs, thus providing a quantitative measure of the two pillars of sustainability - environment and economy (Stanchev and Ribarova, 2016; Caiado et al., 2017). According to the ISO14045 standard (ISO 14045, 2012), eco-efficiency is a relative method to compare different products/systems to identify the most efficient one. In this study, the eco-efficiency method was applied to compare the eco-efficiency performance of WPC and SPC materials. The environmental performance within the eco-efficiency ratio, was evaluated following the life cycle assessment (LCA), as recommended by ISO 14044 (ISO 14044, 2006), while the economic performance was expressed by the monetary costs (ISO 14045, 2012). The analysis followed the cradle-to-factory gate approach. The system boundaries are given in Fig. 1, including all the production stages, the inputs and outputs at each phase. The functional unit selected for the analysis was '1 tonne finished product, packed and ready for dispatch'. The LCA-based Carbon Footprint indicator was used to evaluate the environmental performance of the system. The calculations were performed using the LCA software package SimaPro 8.2.3.0 and the Ecoinvent database v3.2 following ReCiPe midpoint (H) method.

Eco-efficiency was calculated in accordance to ISO14045 (ISO 14045, 2012) as a ratio of the value performance to environmental impact. The value performance was expressed as the value added of the system, or in other terms the price of the final product minus the production costs. Thus, the eco-efficiency performance was calculated by using the following equation:

$$Eco-efficiency = \frac{\text{Value added}}{\text{Environmental impact}} \tag{1}$$

3. Results and discussion

3.1. Mechanical properties of WPC and SPC

The tensile properties of untreated and coupling agent treated WPC and SPC are given in Fig. 2. The untreated WPC and SPC exhibited a tensile stress of 9.10 MPa and 7.49 MPa respectively. The use of MA increased the tensile stress of the WPC and SPC by 88.5% and 81.3% respectively, while the addition of VTMS increased the tensile stress of WPC and SPC by 21.8% and 10.5% respectively. This increase was attributed to significantly enhanced interfacial bonding and more efficient stress transfer across the interface between the cellulosic fillers (i.e. wood particle and sludge cellulose) and polymer matrix with the aid of MA and VTMS coupling agents

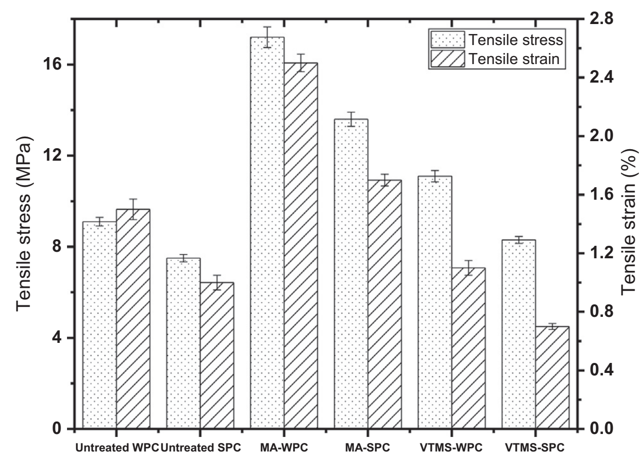

Fig. 2. Tensile stress and tensile strain of WPC and SPC.

(Rao et al., 2018). The intrinsically multifunctional coupling agents were able to covalently bond/crosslink with both hydrophilic lignocellulosic fillers and hydrophobic matrix, such to enhance the wettability, dispersion and distribution of the fillers and the constituent compatibility. The results were the enhanced interfacial adhesion and more efficient stress transfer from the matrix to wood flour or sludge cellulose, thus the improvement of the mechanical properties (Zhou et al., 2017; Rao et al., 2018; Bengtsson and Oksman, 2006). Furthermore, MA coupling agent was also able to enhance the ductility of both WPC and SPC, which was reflected by the increase of tensile strain of the composites (69.2% for WPC and 66.5% for SPC). On the contrary, the application of VTMS resulted in the decrease of the tensile strain of WPC and SPC by 25.1% and 34.5% respectively. This was because the HDPE-VTMS crosslinks had a stiffening impact on the continuous plastic phase (Clemons et al., 2011; Bengtsson et al., 2005), hence the tensile modulus of VTMS treated composites was increased (Fig. 3).

The incorporation of MA into the composites gave rise to a dramatic increment in flexural stress (i.e. 155.4% for WPC and 134.6% for SPC) as shown in Fig. 4. This is consistent with the aforementioned variation of tensile stress, again due to the enhanced interfacial adhesion and bonding. It had been documented that improved interfacial adhesion between wood and plastic enables the stress transfer from the weaker plastic matrix to the stronger wood fibre during loading, thereby improving the strength of the

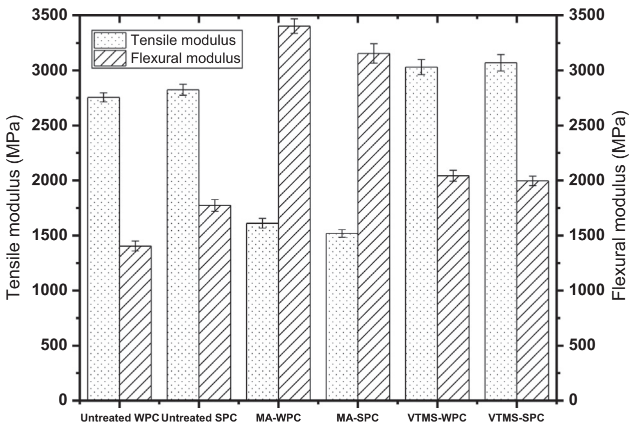

Fig. 3. Tensile and flexural modulus of WPC and SPC.

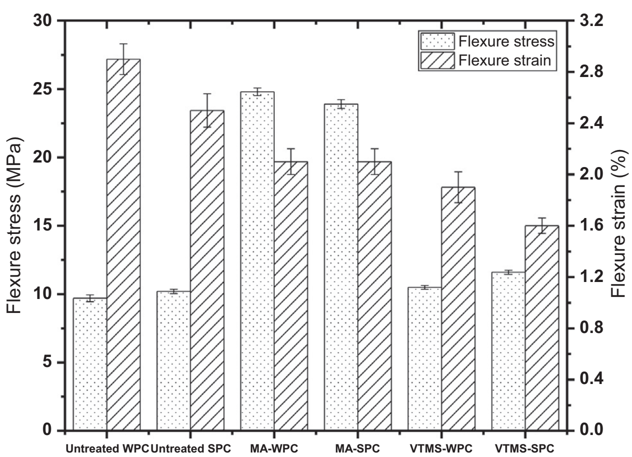

Fig. 4. Flexural stress and flexural strain of WPC and SPC.

crosslinked composites (Bengtsson and Oksman, 2006). In the case of uncrosslinked (untreated) composites, the lack of intimate bonding between wood flour or sludge cellulose and HDPE led to numerous irregularly shaped micro-voids or micro-flaws, making the transfer of stress from the matrix to the cellulosic fillers inefficiently, and partially utilised mechanical properties of the fillers (Adhikary et al., 2008). The flexural stress of VTMS treated WPC and SPC was 8.3% and 13.8% higher than that of the untreated composites respectively, indicating that VTMS should be a less efficient coupling agent than MA in refining the interfacial bonding of the composites. Noteworthy that both MA and VTMS treated composites suggested lower flexure strain than the untreated counterpart. This is in agreement with the findings of previous studies on coupling agent treated WPC, i.e. improving the adhesion between wood or fibres and thermoplastics did not enhance the elongation at break (Adhikary et al., 2008; Sommerhuber et al., 2016). In addition, both VTMS and MA treatments led to an increase in flexural modulus. Earlier studies on coupling agent crosslinking in WPC have shown that the modulus could be affected in both directions, and it was also strongly affected by the filler level and the orientation of the filler and less by the polymer-fibre adhesion (Grubbström and Oksman, 2009). Although SPC showed inferior tensile properties to WPC, e.g. the tensile strength (13.58 MPa) and tensile strain (1.69%) of MA treated SPC were 26.4% and 46.7% lower than those of MA treated WPC respectively, there was no considerable difference in flexural properties between SPC and WPC probably due to the shear stress existed between the layers of SPC. The latter shows that SPC would meet the strength requirements when it is used as decking and fencing profiles, which are the largest application sectors for WPC.

3.2. Thermal property analysis

The TGA curves of untreated and coupling agent treated composites are presented in Fig. 5 along with the extracted data in Tables S2 and S3. It was observed two-step decomposition occurring in all the composites, i.e. first stage from 280 °C to 375 °C and second stage from 440 °C to 495 °C. Both WPC and SPC composites started to decompose at around 280 °C and thus reached the maximum degradation rate at 350–360 °C at the first stage, which was referred to the depolymerisation of hemicellulose, and dehydration and thermal cleavage and scission of glycosidic linkages (e.g. C—C and C—O bonds) of cellulose in wood and sludge cellulose (Manikandan Nair et al., 2001; Mohanty et al., 2006; Mohanty and Nayak, 2006). Although the incorporation of MA and VTMS coupling agents did not appear to affect the initial degradation temperature (around 290 °C) of the composites, the temperature at maximum degradation rate (T_{max}) of coupling agent treated composites was approximately 3 °C lower than that of untreated counterpart (Table S2). However, the weight loss of MA treated composites at first stage (23.2% for WPC and 26.9% for SPC) was around 20% lower than that of untreated composites, which was in agreement with their higher $T_{50\%}$ than the untreated composites as shown in Table S3, indicating that the MA treated composites were more thermally stable. This was probably due to the MA induced crosslinking between cellulosic filler and the matrix, i.e. compared to the uncrosslinked portions, the PE crosslinked wood or sludge cellulose with the aid of MA might degrade with PE at higher temperature (i.e. at second decomposition stage from 440 °C to 495 °C), thus resulting in a higher weight loss at the second stage (Table S2). The crosslinking between the components in the composites also accounted for the aforementioned improvement of the tensile and flexural properties. With respect to the influence of VTMS, the T_{max} at second decomposition stage of VTMS treated WPC and SPC was 6.2 °C and 2.0 °C higher than that of untreated composites respectively, while displaying a similar weight loss (Table S2). However, the degradation temperatures of VTMS treated composites at residual weight 90%, 50% and 20% were slightly lower than the counterparts of untreated composites (Table S3). These results suggested VTMS was not as efficient as MA in terms of enhancing the thermal property of the composites.

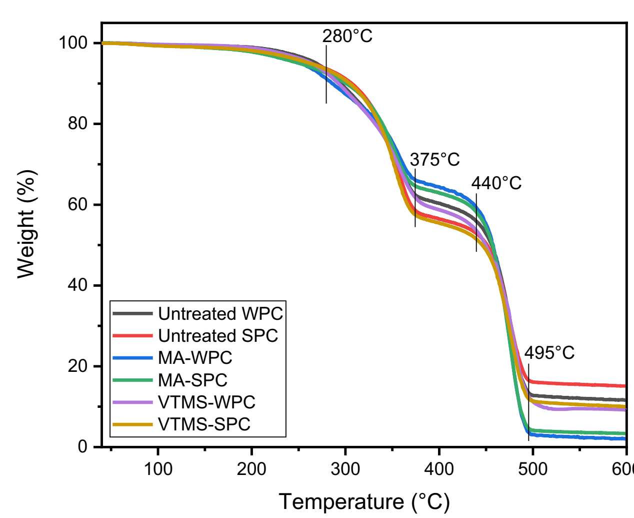

Fig. 5. TGA curves of WPC and SPC.

In addition, WPC was slightly more thermally stable than SPC concerning it demonstrated slightly higher T_{max} at both stages along with higher $T_{50\%}$. Furthermore, SPC had a higher residue than WPC due to its inorganic impurities that possess superior thermal stability.

3.3. Microstructure analysis

The microstructure of the fracture surface of the specimens after tensile tests was examined using SEM, the results are shown in Figs. 6 and 7. The observed cavities or voids on the fracture surface of untreated WPC (Fig. 6a) and SPC (Fig. 7a) along with numerous wood or fibre pull-outs confirmed the poor and week interfacial bonding between the filler and matrix. This also indicated that the failure mode of untreated composites was dominated by pull-out damage rather than fibre breakage and interface debonding (Adhikary et al., 2008; Ou et al., 2010). Although it could still be seen a number of voids on the fracture surface of MA treated composites (Figs. 6b and 7b), the wood particles and cellulose fibres were firmly bonded to the matrix and also well embedded in it with few fibre pull-outs, which substantiated the enhanced interfacial adhesion after the treatment. Furthermore, damaged wood particles were observed on the surface of VTMS treated WPC in addition to well embedded particles (Fig. 6c). This means that the interfacial strength was stronger than the wood particle because the fracture path passed through the wood particle instead of the interface (Grubbström et al., 2010). In the VTMS treated SPC, both the firmly bonded fibres and fibres pull-outs were observed on its fracture surface, which might suggest that its bonding quality is superior to that of untreated counterpart but inferior to that of MA treated composite, resulting in the following sequence of mechanical strength of the composites: Untreated < VTMS treated < MA treated. It can be concluded that different from the untreated composites, the dominant failure modes in MA and VTMS treated composites were matrix fracture, interface debonding, fibre fracture and fibre fibrillation (Figs. 6b, c, and 7b, c) due to the significant improvement of bonding quality between the filler and matrix (Ou et al., 2010).

3.4. Eco-efficiency assessment

In order to conduct the eco-efficiency analysis for WPC and SPC materials, primary data on the materials and process energy inputs were collected from a construction material company in England in 2018 to develop the materials and energy balances in the production process (Fig. 1). The only waste stream in the processing

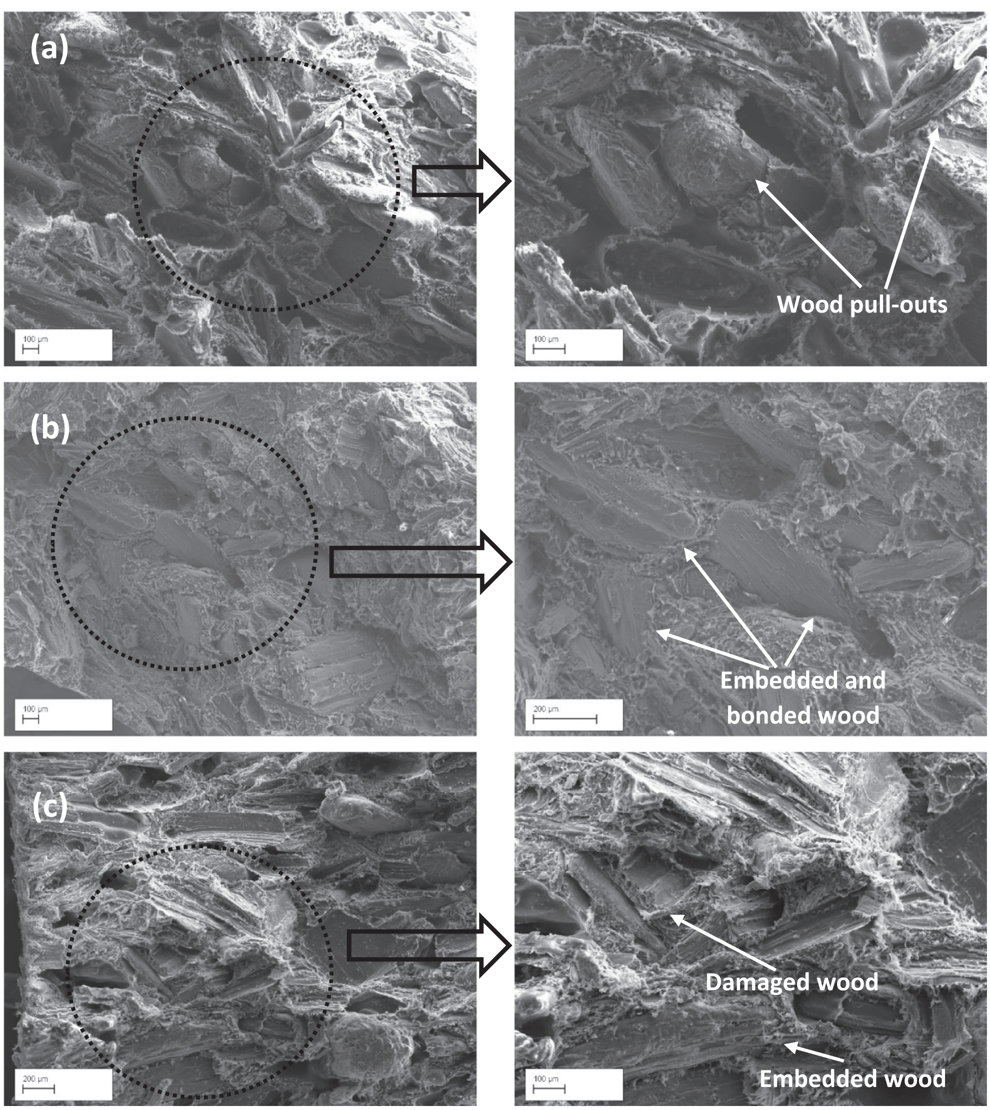

Fig. 6. SEM images of the fracture surface of WPC (a: untreated; b: MA treated; c: VTMS treated) after tensile tests.

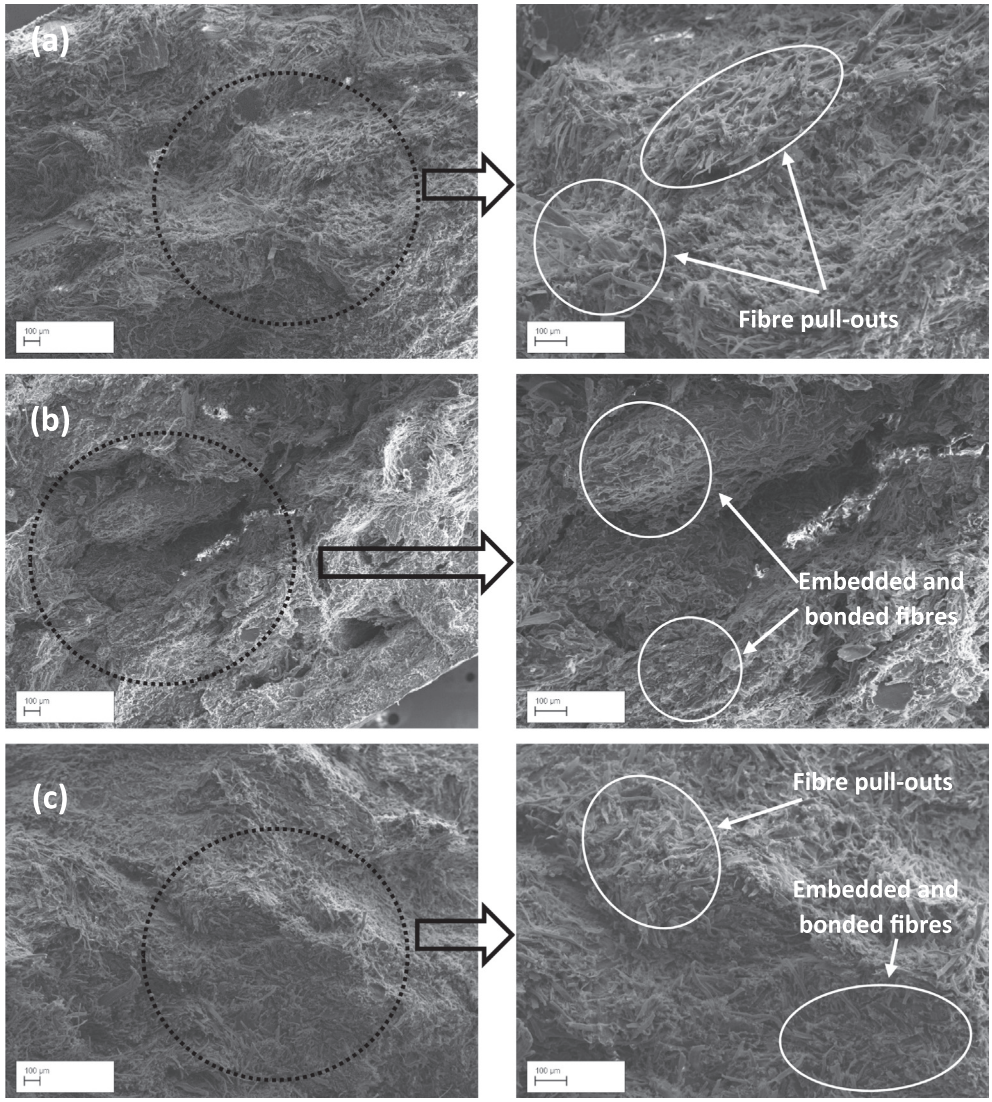

Fig. 7. SEM images of the fracture surface of SPC (a: untreated; b: MA treated; c: VTMS treated) after tensile tests.

system was generated from the dryer, where a very small proportion of cellulosic filler was lost (i.e. 0.45% of wood flour and 0.63% of sludge cellulose). Thus, the impact of the generated wood and cellulose waste was not considered in the analysis.

The energy demand at different production stages of WPC and SPC is given in Table S4. SPC drying requires 14% more energy than the respective WPC drying due to the higher moisture content in sludge cellulose. However, its extrusion process shows considerable energy savings (~10 kWh/t), which results in total energy savings of 2.6% compared to the WPC.

The unit prices of the raw materials and additives used in the production were also collected from the construction material company. The results of the economic value performance evaluation for WPC and SPC are summarised in Table S5.

Table 2 shows the results from the comparison of the economic, environmental and eco-efficiency performance of the WPC and SPC materials. The embodied Carbon Footprint of wood flour and sludge cellulose was assumed as zero, since both materials were derived from waste streams. The LCA results showed that SPC had a slightly lower Carbon Footprint compared to WPC, due to the minor reduction of the energy requirements in its production. In terms of economic value performance, the production cost of SPC was significantly lower than the respective one of WPC (approximately 15%) as a result of the lower price of the sludge cellulose. The latter showed the higher value added of SPC. The eco-efficiency indicators for both products were calculated based on the Carbon Footprint and value performance results. Assuming that two products serve the same function, SPC product was more

Table 2
Results of the eco-efficiency assessment.

Composite	Environmental performance	Economic performance			Eco-efficiency (£/kg CO_2eq)
	Carbon footprint (kg CO_2eq/FU)	Product price (£/FU)	Costs (£/FU)	Value added (£/FU)	
WPC	1,338.9	£3,800	£851	£2,649	2.23
SPC	1,333.9	£3,800	£722	£2,778	2.26
Difference	−0.37%	–	−15.17%	4.87%	5.26%

eco-efficient than WPC by 5.26%. The latter suggested that from both environmental and economic points of view, cellulosic sludge can be a suitable and prospective alternative to wood flour in WPC production. The valorisation pathway of cellulose fibres recovered from wastewater to WPC industry has not been studied yet. There are only a few pilot studies on recovery of cellulose fibres from wastewater, indicating that the technology is not yet mature on a full-scale perspective (Papa et al., 2017). Key factors to the successful upscaling of the technology are the market potential, regulatory barriers as well as consumer acceptance. However, the use of recovered sludge cellulose in WPC industry could potentially boost the circular economy transition of WWTP to wastewater resource recovery facilities.

4. Conclusions

Sludge cellulose recovered from WWTP was firstly used to develop compatible SPC materials with the aid of MA and VTMS coupling agents. The mechanical and thermal properties, manufacturing costs, and environmental impact of SPC were analysed to determine the substitution potential of sludge cellulose in WPC. The use of MA and VTMS coupling agents significantly improved the tensile strength and flexural strength of both WPC and SPC materials due to enhanced interfacial bonding and more efficient stress transfer through interface, which were confirmed by the microstructure analysis. MA treated composites exhibited higher thermal stability by showing less weight loss and higher degradation temperature ($\mathbf{T_{50\%}}$). There was no considerable difference in flexural properties between SPC and WPC, suggesting that SPC would meet the property requirements when it is used to replace WPC as decking or fencing profiles. Finally, cradle-to-gate input-output inventory based analysis of economic, environmental impact and eco-efficiency demonstrated that SPC had 15% lower manufacturing costs and it was 5.26% more eco-efficient than WPC. The use of cellulose fibres recovered from wastewater in WPC industry has the potential to close the cellulose material loop and develop a new market that can boost the circular economy transition of WWTP.

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Appendix A. Supplementary material

Supplementary data to this article can be found online at https://doi.org/10.1016/j.wasman.2019.08.037.

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