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The Impact of Primary Sludge on the Physical Features of High-Density Polyethylene (HDPE) Composites

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Abstract: The cost-efficient reutilization of byproduct materials is a significant global goal, contributing towards the sustainable use of resources. In this study, the effects of including primary sludge in composite materials on their physical performance are examined, in order to achieve more effective reuse. The studied materials were made from high-density polyethylene (HDPE), anhydride-grafted polyethylene (MAPE), lubricants, and either wood flour from spruce (*Picea abies*) or primary sludge from the side-stream of forest industry processes as a filler. The materials were compounded by agglomeration, followed by manufacturing with a conical twin-screw extruder. The physical properties of the materials were characterized by water absorption and thickness swelling tests; furthermore, impact strength was characterized after the stress of a cyclic freeze-thawing test. The elemental compositions of the materials were also analyzed by scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy (SEM-EDS). Primary sludge, as a component in the structure of the composite material, resulted in a significant improvement of moisture behaviors in the water absorption and thickness swelling tests. The identified results demonstrate that primary sludge is a technically applicable material for utilization in composite materials.

Keywords: primary sludge; composite; water absorption; thickness swelling; freeze-thaw cycling

1. Introduction

Industrial byproducts may serve as valuable raw materials in applications, and their utilization as raw materials can contribute, simultaneously, to the idea of a circular economy. The utilization of byproducts reduces the exploitation of primary resources and waste disposal, while simultaneously creating economic growth. A good example of such a byproduct is sludge from the forest industry, which could be used more efficiently in the future.

Primary sludge is the residual solid generated by effluent treatment. The main effluent treatment process used for pulp and paper mill effluents consists of primary and secondary treatment processes. In the primary treatment, the effluent is mechanically processed (sedimentation, flotation, or filtration) to remove fibers and suspended solids. Solid matter is settled by gravity in a clarifier, thus generating the primary sludge. The effluent treatment process is further continued with a secondary treatment, where dissolved organic compounds are removed with biological methods. The most usual method is an activated sludge process, where microbes oxidize the organic matter into biomass, carbon dioxide, and water. After biodegradation, the biomass and water are separated, typically using sedimentation tanks. Biosludge, also referred to as secondary sludge, is generated as a result. Primary sludge and secondary sludge are then mechanically dewatered, either together or separately, before further handling or disposal [1–5].

At present, common disposal methods for forest industry sludge are incineration in the bark boiler or composting. In some cases, sludge may still be landfilled, which is very restricted or even forbidden in many countries [5,6]. According to the European Commission Waste Framework Directive [7], waste should be reused or recycled before recovering for energy. Various national regulations will restrict waste disposal. For example, in Finland, the total organic carbon (TOC) content of landfill waste cannot be more than 10% (Finnish National Waste Directive 331/2013), meaning that primary and secondary sludges are not allowed in landfills. In addition, waste that is landfilled has been subjected to taxation in many countries, and tightening environmental limits have forced countries to take actions towards new, more sustainable solutions.

Primary sludge consists of wood fibres, cellulose, hemicellulose, lignin, pigments, resin compounds, and ash. Its fibre content varies between 40 and 70%, depending on the processes within the production site. The solid content of the primary sludge can be 40–50% after the mechanical dewatering [8–10]. Primary sludge contains organic matter, which should be recovered and utilized as a resource, instead of as an energy recovery option. Furthermore, there are also challenges concerning the incineration of the sludge. The main drawback of dewatered sludge is the low net caloric value, due to high content of water. In addition, the incineration of wet material increases the flue gas volume in the boiler [5]. However, incineration is a common means of sludge disposal, as it reduces the waste volume and, thus, reduces landfill costs. In addition, the generated ash can be utilized for the production of light-weight aggregates, bricks for use in construction, or even as forest fertilizer [8].

Various studies have focused on the utilization of solid wastes from the forest industry as a raw material resource, but not many have focused on primary sludge. Malaiskiene et al. [11] have evaluated the impact of using primary sludge on the properties of a cement mixture. It was discovered that small quantities (max 5%) could be used without deteriorating the strength properties of the mortar too much. Fiber-containing sludge has benefits in producing bricks. For example, it has been shown that increased fiber content enhances the porosity of the matrix and, thus, leads to lighter bricks, while saving fuel in the oven and decreasing the cooking time [12]. The use of paper industry sludge in stone mastic asphalt as a fiber additive has also been studied. Four different sludges were examined, and the de-inking sludge was found to be the most suitable. However, all four tested sludges passed the general specifications for medium- and heavy-traffic road pavement, according to the Department of Public Works and Highways [13]. In addition, other alternative raw materials might be useful in wood-based composites, such as wheat husk [14].

A certain application for sludge might be as a composite, which is a combination of two or more materials resulting in more favourable performance than that of either of the individual components used on their own. The composite consists of a continuous phase matrix and a reinforcing phase, which is usually a fibre or a particulate. One example of this is the wood–plastic composite (WPC), wherein a polymer matrix is reinforced with a wood component in particle form [15]. Soucy et al. [16] studied the potential of paper mill sludge as a raw material for a wood–plastic composite, in which the sludge consisted of primary and secondary sludges in different ratios. Based on the results, the primary sludge had a reinforcing effect on the WPC, while the secondary sludge had negative impact on the physical and mechanical properties of the composite. Lahtela et al. [17] found that replacing wood flour with primary sludge increased the flexural strength, hardness, and impact strength of the composite. The above described supports the hypothesis that primary sludge can act as a raw material for WPC from the aspect of physical features.

In this study, certain physical properties, such as water absorption, thickness swelling, and effect of aging on the mechanical properties of WPC are compared in a composite where the wood material is replaced by primary sludge from the forest industry. The objective of the present study is to determine the functionality of primary sludge in the composites and how its use affects the physical properties of the produced composites.

2. Materials and Methods

2.1. Materials

High-density polyethylene (HDPE), RecyPE HDPE 8 blue (L&T Muoviportti, Merikarvia, Finland), with density 0.960 g/m^3 and melt mass flow rate 8 g/10 min (190 °C/2.16 kg), was used as the composite matrix in the experiments. The coupling agent was maleic anhydride-grafted polyethylene (MAPE), Fusabond E226 (DuPont, Geneva, Switzerland), and Struktol TPW 113 (Struktol, Stow, OH, USA) was used as the lubricant. The filler of the composite was either wood flour or primary sludge (Pr-Sl). The wood flour was prepared from a dried spruce species (*Picea abies*), which was produced by hammermilling and sieving (20-mesh). The Pr-Sl originated from the side-stream of the forest industry, which was treated to achieve a more solid form, as follows: Effluents from a paper mill, a pulp mill, and woodhandling were treated in a wastewater treatment plant (WWTP). Primary clarification was performed in the WWTP, where the Pr-Sl was directed into a mixing tank and collected before mixing with secondary sludge. The solid content of untreated Pr-Sl was 4.3%, followed by screening for bigger particles and flocs, after which the screened Pr-Sl was pressed with a laboratory membrane filter apparatus (Outotec Larox MFP 0.3, Lappeenranta, Finland) equipped with a polypropylene filter (ASKO T50, Lappeenranta, Finland). After the treatment, the solid content of primary sludge was 42.7%. The compositions of the studied composites are presented in Table 1.

Table 1. The component (high-density polyethylene (HDPE), wood flour (WF), primary sludge (Pr-Sl), maleic anhydride-grafted polyethylene (MAPE), and lubricant) amounts of the composite. Amount shares are given in percentage (%), based on gravimeter reading.

Composite	HDPE	WF	Pr-Sl	MAPE	Lubricant
Reference	50	44	-	3	3
Pr-Sl/HDPE	50	-	44	3	3

2.2. Work Methods

The components were agglomerated prior to extruding with an agglomeration apparatus consisting of a PLASMEC TRL 100/FV/W turbomixer (Lonate Pozzolo, Italy) and a PLASMEC RFV-200 cooler (Lonate Pozzolo, Italy). After agglomeration, the components were again produced by hammermilling through a 4.00 mm sieve. The materials were manufactured to a profile using a conical counter-rotating twin-screw-type extrusion machine (CE 7.2 FE—Hans Weber Maschinenfabrik Gmbh, Kronach, Germany). All components were fed into the extruder through the gravimetric feeding system. The reference material was extruded through an approximately 3.00 mm thick flat die, while the Pr-Sl/HDPE material was extruded through a rectangular die, as depicted in Figure 1.

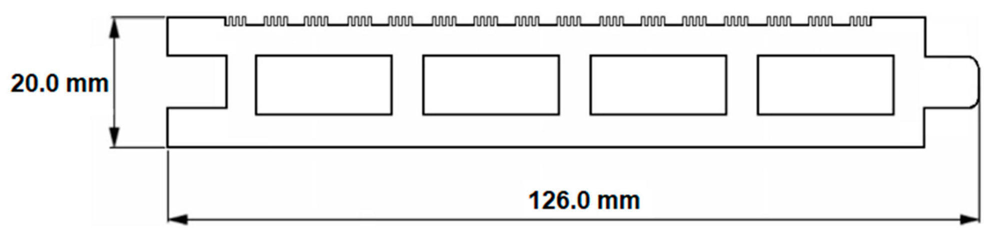


Figure 1. Schematic of the hollow-shaped decking board composite profile.

The reference material was processed at a barrel temperature of 139 °C (average of seven measurements in the mixing zone), and the melt was processed at 140 °C with a pressure of 2.3 MPa. The corresponding parameters for Pr-Sl/HDPE were 171 °C, 174 °C, and 4.0 MPa, respectively. The feed rate was 20 kg/h for both materials, and the used screw speeds were 13 (reference) and 14 (Pr-Sl/HDPE) rpm.

In this work, the samples for the impact strength, water absorption (WA), and thickness swelling (TS) experiments were prepared by cutting from the extruded profile. The test methods were based on the recommendations of standard EN 15534-1:2014 + A1:2017 [18].

2.2.1. Moisture Properties

The WA and TS of materials were determined by measuring the weights and dimensions of the samples, based on the procedure of standard EN 317 [19]. The results were calculated using the following equations:

$$\text{WA}(\%) = (m_t - m_0)/m_0 \times 100 \tag{1}$$

$$\text{TS}(\%) = (t_t - t_0)/t_0 \times 100 \tag{2}$$

where m_0 and m_t are the masses of the sample before and after immersion, and t_0 and t_t are the thicknesses of the sample before and after immersion, respectively. The presented values are average values consisting of twenty square-size samples whose side length was 50 mm. The thickness of samples was based on the nominal thickness of the extruded profile, which was measured by micrometer apparatus.

2.2.2. Impact Test After Freeze-Thawing Cycles

The Charpy impact strength for the unnotched samples was measured with a Zwick 5102 model impact tester, in accordance with standard EN ISO 179-1 [20], using method ISO 179-1/1fU. The impact strength was measured with and without a cyclic freeze-thawing cyclic treatment, according to the standard EN 15534-1:2014 + A1:2017 [18] where samples are exposed in a water bath, freezing cabinet (Electrolux, EC4230A0W2/BNI425), and drying cabinet (Gallenkamp, Hotbox Oven with fan, Size 2). The performed tests for cyclic treatment were carried out with eight sample replicates for both types of composite, for which dimensions were 80 mm × 10 mm × 4 mm (length, width, and thickness, respectively). The results are presented as average values.

2.2.3. SEM Analysis

The surface morphology of the samples was studied with SEM analysis. A scanning electron microscope coupled with energy-dispersive X-ray spectroscopy (SEM-EDS, Hitachi SU3500, Tokyo, Japan) was used to investigate the cross-sectional surfaces (10 mm × 4 mm) of the impact strength samples. SEM analysis was performed with a few replicates for both types of composite, which were represented as average values from the impact strength tests. Elemental analyses of the samples were performed with EDS (Thermo Scientific, Waltham, MA, USA), which is known as a nondestructive analytical method, and it may confirm the presence or absence of elements.

3. Results

3.1. Material Characterization

The structure of the reference material was more porous, compared to the Pr-Sl/HDPE materials, as can be seen in Figure 2. The porosities of the structures may be explained by the densities of the materials, which were measured from the 20 samples after processing. The average density values of primary sludge and the reference material were 1.14 g/cm^3 and 0.96 g/cm^3, respectively.

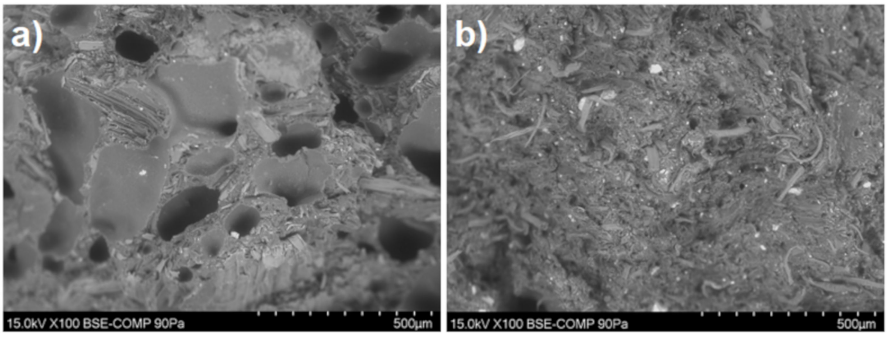


Figure 2. Scanning electron microscope (SEM) images of the structure of reference (**a**) and Pr-Sl/HDPE (**b**) materials, at a magnification of ×100.

An element analysis by SEM-EDS identified the elemental composition of the Pr-Sl/HDPE materials, which are presented in Table 2 as an average value from three samples. The observed metal elements in the materials are as follows: Sodium (Na), magnesium (Mg), aluminium (Al), silicon (Si), potassium (K), calcium (Ca), iron (Fe), zinc (Zn), molybdenum (Mo), and chlorine (Cl). The most general metal elements were Mg, Al, and Si, whose weight percent (wt %) shares varied between 1.40 and 3.95 of the whole mass of the sample. These elements were found in all studied samples. The other elements (Na, Cl, K, Ca, Fe, Zn, and Mo) were not included in every studied sample, as some of the studied samples had zero content of a certain element. For example, chlorine was included only in one sample with a low content (0.26 wt %).

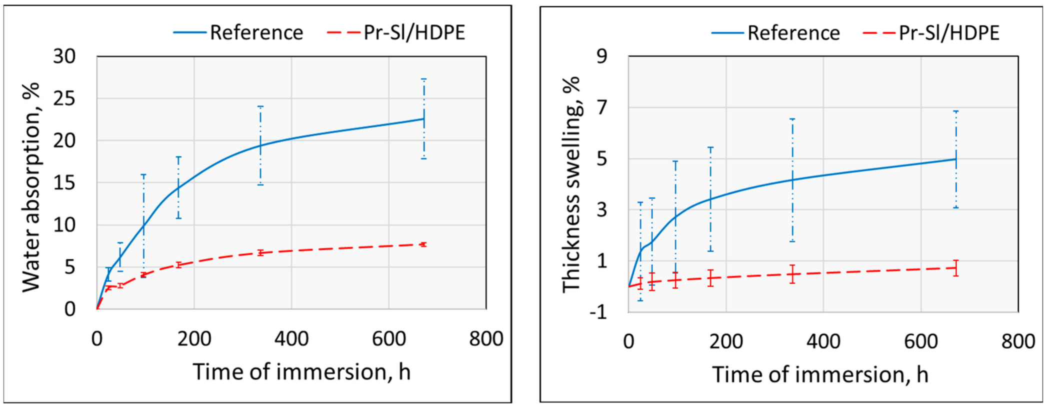
Table 2. The elemental composition of the Pr-Sl/HDPE materials. Amount shares are given as an average of weight percentage (wt %) from the whole mass of the sample.

Element	Avg. (wt %) [1]	Sd [2]
Sodium (Na)	0.47	0.39
Magnesium (Mg)	1.40	0.82
Aluminium (Al)	1.98	1.07
Silicon (Si)	3.95	2.38
Chlorine (Cl)	0.09	0.12
Potassium (K)	0.05	0.07
Calcium (Ca)	5.92	8.37
Iron (Fe)	0.35	0.49
Zinc (Zn)	0.06	0.09
Molybdenum (Mo)	1.03	1.46

[1] Average, [2] Standard deviation.

3.2. Moisture Properties

The moisture properties of the tested materials are presented as a scatter chart in Figure 3, in which smooth lines have been added between the data points. In addition, standard deviations are added with error bars. The water absorption and thickness swelling of the reference material increased significantly, as compared to the Pr-Sl/HDPE material. The increase was intense during the first 200 h of immersion. Over the 28 days of immersion in the water bath, the reference material had WA of 22.58% and TS of 4.97%, on average. Correspondingly, WA reduced by over 65% and TS reduced by over 85% for Pr-Sl/HDPE material. In particular, the TS of the Pr-Sl/HDPE material was minor, only 0.72% after 28 days of water immersion. The standard deviations of the measured average values were considerably different between the materials. The Pr-Sl/HDPE composite had a restrained deviation in the WA and TS tests, while the reference had a wide deviation, especially in the TS test.



(a) (b)

Figure 3. Water absorption (**a**) and thickness swelling (**b**) of the studied materials. The uniform line represents the reference material and the discontinuous line represents the Pr-Sl/HDPE material. The results consist of average values, with error bars describing the standard deviations.

3.3. Freeze-Thawing Durability of Materials

Table 3 shows the effects of the cyclic freeze-thawing test on the impact strengths of the materials. The results after three cycles are average values from eight measurements, while the values before the cycles were derived from twenty measurements. The results show that the variation of freeze-thawing reduced the impact strength of the reference material, while, with the primary sludge (Pr-Sl/HDPE) material, the impact strength increased slightly.

Table 3. The effects of the cyclic freeze-thawing treatment on impact strength.

	Reference Before Cycles	**Reference After 3 Cycles**	**Pr-Sl/HDPE Before Cycles**	**Pr-Sl/HDPE After 3 Cycles**
Impact Strength (kJ/m^2)	4.25	3.09	6.37	6.91
Standard deviation	±0.51	±0.36	±0.66	±0.81

The influence of the cyclic treatment is also shown in Figure 4, which presents SEM images from cross sections of the tested impact strength samples, taken before and after cyclic stress. The reference material was more porous compared to primary sludge (Pr-Sl/HDPE) material. Therefore, the reference material absorbed a higher amount of water into its structure. The water in the structure expanded as it froze, causing structures to fail and thus reducing the impact strength.

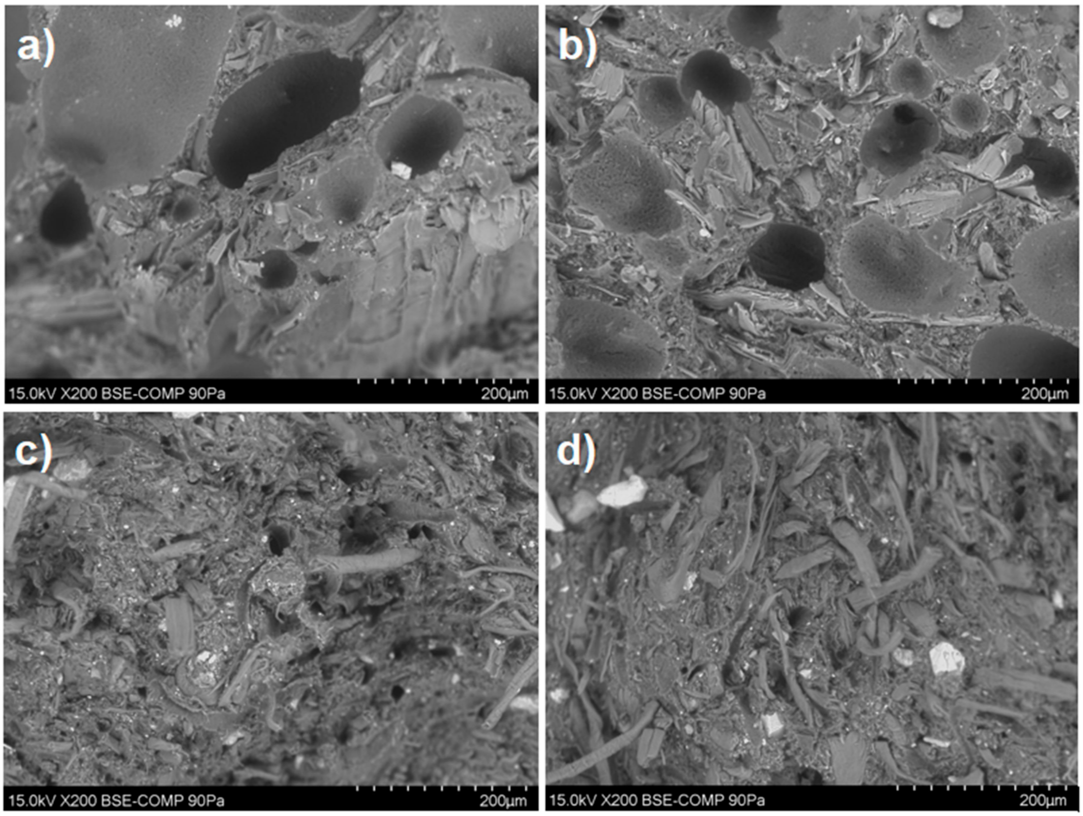


Figure 4. SEM images of the cross sections of impact strength samples at a magnification of 200×. Images (**a**,**b**) (top) are the before and after images for the reference sample, and images (**c**,**d**) (bottom) are the before and after images for the Pr-Sl/HDPE sample. Images (**a**,**c**) (left) are before the cyclic treatment, and images (**b**,**d**) (right) are the samples after three cycles of the cyclic treatment.

4. Discussion

Utilization of primary sludge, instead of wood flour, induced a tighter structure in the composite material, as can be seen from the density results and the SEM images. Previously, it has been found (with another material) that pores and their quantities increased with an increased content of primary sludge [11]. Otherwise, denser materials require higher treatment parameters in the processing steps, which may not be the most economical option. A higher treatment temperature also exposes material to thermal degradation. The elemental analysis of the Pr-Sl/HDPE material showed small amounts of metal elements, which may have originated from the paper production process. Calcium carbonate and kaolin are typically used as a filler in base paper and as a pigment in coating color used for paper coating, respectively. The bleached kraft pulp process includes several bleaching sequences to attain a target brightness level. One of the typical bleaching chemicals used is chlorine dioxide, which could explain the chlorine detected in the elemental analysis.

The large deviation in the WA and TS results of the reference material is congruent with a previous study of similar materials [21]. Water absorption has been shown to act as an indicator for composite porosity [22], which was also found in this study. A previous study showed that, by replacing wood materials in the structure of WPC with another material, moisture effects were reduced [23]. Generally, this has been explained by the hydrophilic nature of wood materials; considered in more detail, its components (i.e., cellulose, hemicellulose, lignin, and extractives) also have an influence on the behavior of the material in the presence of moisture. For example, the removal of hydrophilic extractives has been shown to increase the interfacial bond between the matrix and fillers [24]. WA and TS were increased with the inclusion of hydrophilic materials, such as the reference material, while more hydrophobic materials could form a protective barrier against moisture absorption [25]. Less hydrophilic materials, thus, can enable better compatibility between the components and limit water penetration of the interface, consequently decreasing water absorption [26]. The presence of wood flour in the structure of a composite makes the material more susceptible to moisture, compared to the corresponding material including primary sludge. One way to restrict absorption while using hydrophilic components is to use a smaller particle size, which decreases the maximum water absorption. Larger particles have been shown to create more space between the components, which creates more easily accessible space for water absorption [27].

Moisture properties have an important effect on the mechanical functionality of WPC. For example, swelling causes stress on the interfacial bonding of WPC's components, which weakens the strength of material. The bond between fiber and polymer matrix is disrupted when the composite is in contact with water. Water molecules form hydrogen bonds with cellulose after long-time immersion in water, which may impair the mechanical properties of the composite [28]. As mentioned previously, the moisture properties were impaired by a shorter fiber length, while the mechanical properties were increased with increasing fiber length [29]. One option for improving the mechanical properties is to add the coupling agent MAPE [30]; however, hydrophilicity also has an effect on the mechanical features. The compatibility of an HDPE matrix and wood filler has been enhanced by decreasing hydrophilic content [31]. Our results have shown that primary sludge can improve the impact strength property of a composite, which the freeze-thaw cycle could not weaken. Sludge, as a component of a composite, appears to be quite stable, in terms of impact features, because the effects of thermos-treatment have been shown to leave them nearly unchanged [32]. Better moisture resistance features might be caused by such improved properties after cyclic treatment, in which the stress of freezing and thawing did not have an obvious effect.

5. Conclusions

In this study, we investigated the effects of including primary sludge on the physical properties of a HDPE composite, consisting of 50% HDPE and 44% primary sludge or wood flour, together with 3% of both a coupling agent and a lubricant. The use of primary sludge in the composite resulted in a stronger structure. In conclusion, based on the physical material feature tests we performed, primary sludge from the forest industry can act as part of the composite structure. Moreover, primary sludge, when used as the matrix in the composite structure, significantly reduced its exposure to moisture. In addition, a cyclic freeze-thawing test did not have a degenerative effect on the material. On the contrary, it slightly increased the physical properties, in the case of the measured feature (impact strength). The achieved results demonstrated that primary sludge is an applicable component in composite materials, from a technical viewpoint. However, there remain properties that still require further investigation; for example, the influences of life cycle should also be assessed.

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