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MECHANICAL RECYCLING OF WOOD PLASTIC COMPOSITES MOULD

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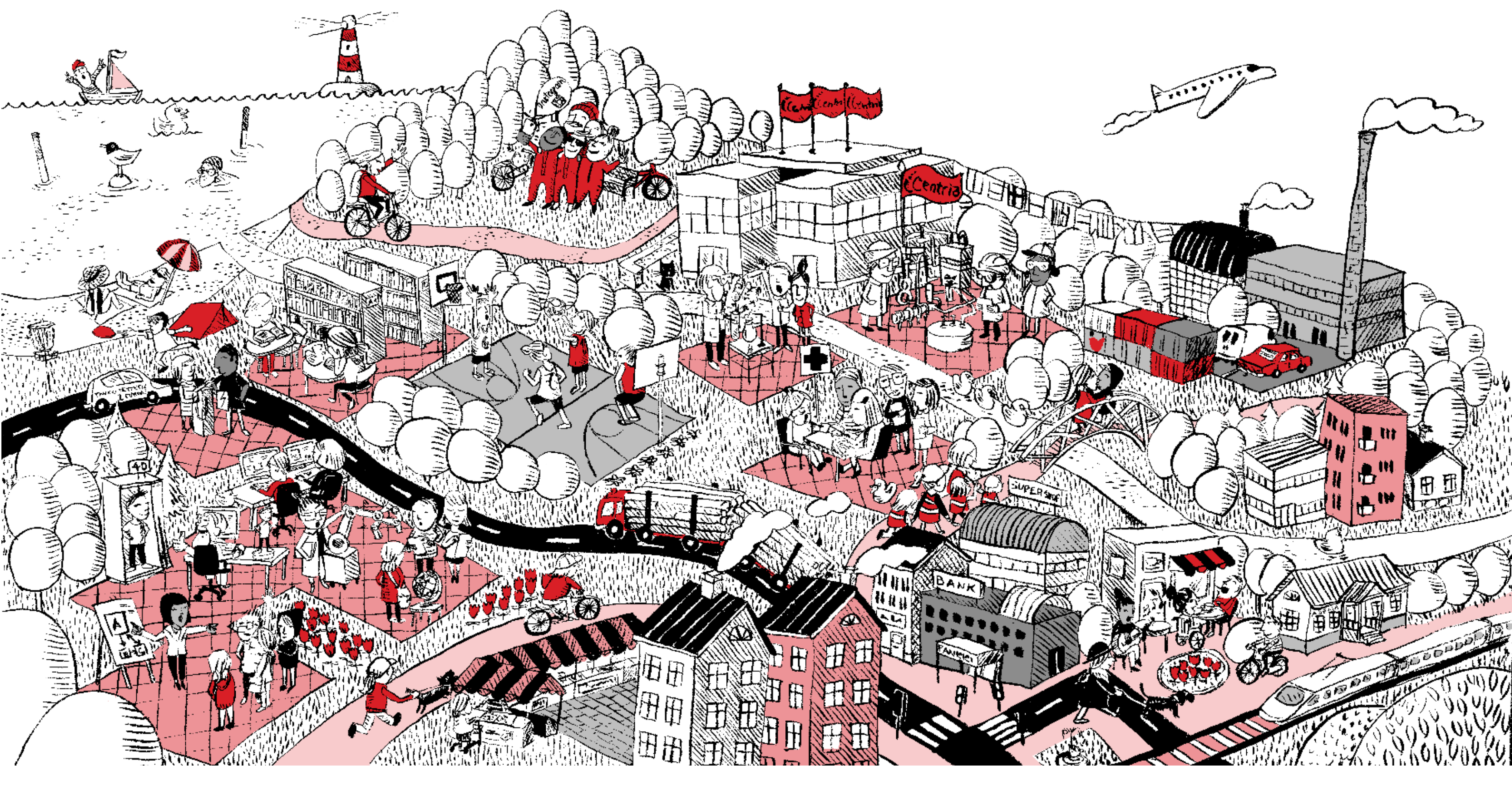

ABSTRACT

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With the development of Fused Granular Fabrication 3D-printing technology, wood plastic composite (WPC) has been researched and applied in many processes such as mould manufacturing. The 3D-printed WPC waste has also many potentials to recycle and bring the end-of-life product back to the mould industry. To contribute on Circular Economy, the recycled WPC mould can be an alternative solution to non-recyclable thermoset mould.

This thesis studied about the feasibility of recycling WPC moulds by focusing on the effect of multiple recycling steps on the physical and mechanical properties of WPC. To represent the 3D-printed mould, the waste created during the 3D-printing trials for producing mould was selected for further investigation. In the first recycling stage, the 3D-printed waste was grinded, and compression moulded to make composite plates. The same recycling steps were repeated four more times. The mechanical properties of grinded powder and composites of each recycling stage were tested and discussed. The result of melt flow index shows an increase, which means the easier draining through the nozzle of 3D-printer. Density illustrates the nearly same results of reference plate and plate of cycle 1; however, it rises significantly from recycling cycle 2, because of increasing tooling gelcoat content. Tensile properties of two first recycling process are stable due to improvement of fibre dispersion. By optical microscopy, the coating particles content show high effect on weaker adhesion between components of the composite. Therefore, tensile properties reduce considerably from recycling cycle 3.

Key words
Compression moulding, mould manufacturing, recycling, wood plastic composite

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ABBREVIATIONS

WPC	Wood Plastic Composite
FGF	Fused Granular Fabrication
MAPP or MAgPP	Maleic Anhydride-grafted Polypropylene
MAPE	Maleic Anhydride-grafted Polyethylene
PP	Polypropylene
HDPE	High Density Polyethylene
PE	Polyethylene
ABS	Acrylonitrile butadiene styrene
PVC	Polyvinyl chloride
PS	Polystyrene
MEKP	Methyl Ethyl Ketone Peroxide ($C_8H_{18}O_6$)

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1 INTRODUCTION

Industrialization is usually the response of country development; however, waste is also created from industrialization. A large amount of waste has been produced every day in the world, which leads to a lot of problems about environment associated to disposal, human and animal health, and pollution. Additionally, storage in land area and marine area for industrial waste is limited. The disposal waste and protection of natural resources are always critical issues. Thus, industrial solid waste utilization has been saving the natural resources, energy source, environment and also bringing financial benefits (Jassim 2016).

Wood plastic composite (WPC) has been seen as an environmentally friendly material for a long time. WPC has been studied with many trials based on the concept of a Cradle-to-Cradle concept to recycle at the end of wood fibres and plastic life cycle and utilize in many industry applications (McDonough & Braungart 2002). WPC is a composite with growing applications which are possible to use a mixture of wood waste and recycled plastic. Therefore, future sustainable usage of WPC is assured by a part of preventing depletion of virgin materials (Youngquist, Myers & Harten 1992). In order to contribute on the development of circular economy, due to high attention for an alternative to traditional polymeric materials, recycling of WPC also needs to be studies (Krause, Sauerbier, Koddenberg & Krause 2018).

In the moulding industry, moulds are made after a desired product is designed (Vaughan 2002). The materials currently used in mould building are non-recyclable and most often they end up in landfill sites or are incinerated. According to the principles of circular economy, incineration of a product is the least preferred end-of-life option. Utilization of recyclable material like WPC for mould building is an environment friendly approach. Currently the WPC is used in many industries like automotive, consumer goods, and in building and construction. Moreover, with technology development, moulds can be made by three-dimensional-printing (3D printing) that is designed with specific shapes in a computer. The studies about the recycling process of WPC moulds can help bring the end-of-life product back to the mould industry, also the environment and financial benefits.

The objective of this research is to study the feasibility of recycling 3D-printed WPC moulds. The properties of the recycled materials and tensile properties of from compression moulded test specimens are studied by using waste materials from 3D printer. Recycling process of wood plastic composite mould was studied in five cycles of compression moulding method. From the studies, it is assumed that recycling can be an opportunity to help save energy, labour and raw materials to produce the moulds.

2 CURRENT SITUATION OF MOULD PRODUCTION

In many industries, moulds play a key role in creating uniform products through moulding process. Moulding is a manufacturing process that shapes liquid or pliable material by using a mould. A wide amount of product parts such as automotive, appliance and electrical devices are produced by the moulding process (Shrivastava 2018).

Steel has been used as the most popular material for mould manufacturing. However, currently, several different materials and composites have also taken over parts of their market (Vasco, Capela, P & Granja 2007). Besides steel, moulds can also be made from thermoset plastic with lower cost and lighter weight. The material is chosen by several criteria depending on the products. Thermoset plastic satisfies the requirement for moulds that are resistant to corrosion, heat, catalyst, ultraviolet state, insoluble in organic solvents and not able to re-melted or re-formed after initial forming (Wegman & Van Twisk 2013). However, when the mould becomes obsolete or broken, it can be only grounded, energy recovery or landfilled.

There are many researches about recycling solution for thermoset moulds, such as using grinded thermosets as a fibre or filler replacement material, however, there are still limitations (Goodship 2012). Moreover, according to Waste Framework Directive 2008/98/EC for managing and disposing of waste, the first step in the five-step "waste hierarchy" is "Prevention", that means "what has not been produced, does not have to be dealt with" (The European Parliament and The Council 2008); (El-Haggar 2007). Therefore, the requirement to replace using thermoset plastic in industry is more important.

Because of recycling issue of thermosetting plastic, alternative sustainable materials are explored to reduce the environmental impacts from industrial production. Different from thermoset plastic, thermoplastic can melt at specific temperatures, be reshaped and be suitable to reprocess (Asim, Jawaid, Saba, Ramengmawii, Nasir & Sultan 2017); (Ashori 2008). Therefore, wood plastic composite is one of good options for main material used in mould, because it includes wood fibre, thermoplastic and small amount of additive. WPC mould can be first printed by Fused Granular Fabrication 3D printer. Rough surface of raw mould is sanded to desired degree. Then, the tooling gelcoat is applied on the surface by painting, then cured, and polished to finish moulds. The WPC mould example is shown in FIGURE 1. The WPC mould is better solution for mould making because not only it can be made from waste of wood production and thermoplastic, but also it is environmentally friendly.

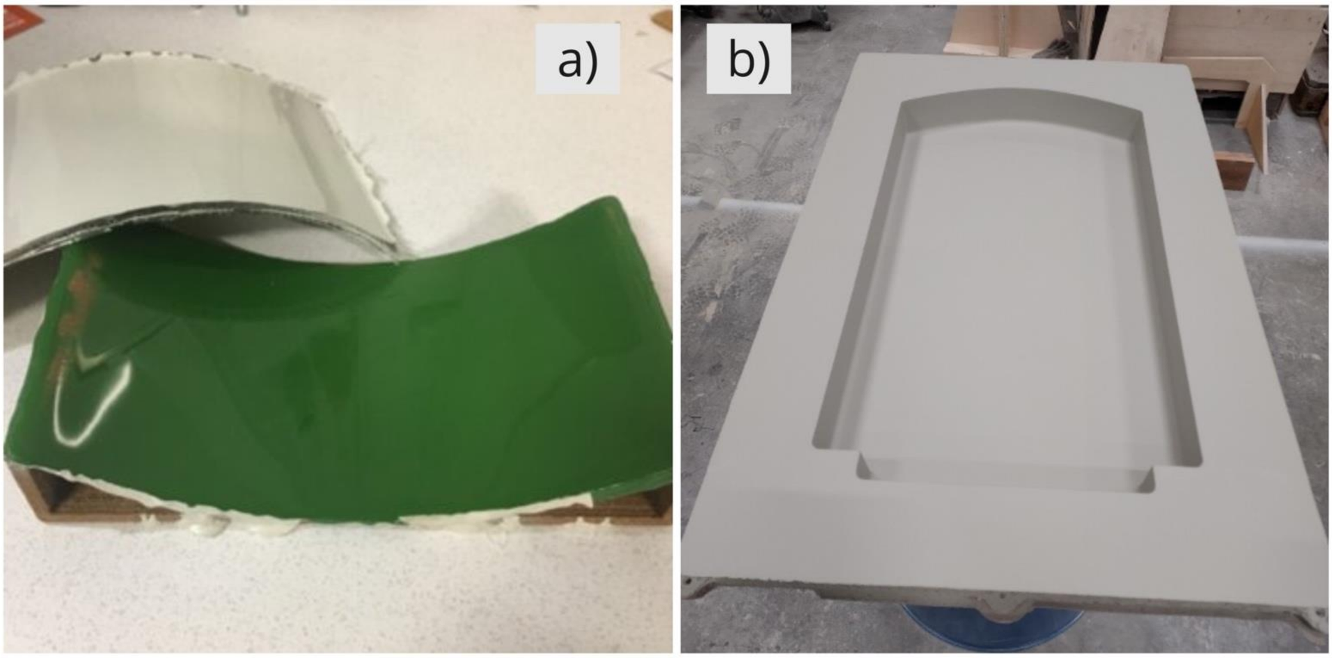


FIGURE 1 Proof of concept WPC mould produced by fused granular fabrication method: a) green surface 3D-printed mould and fibre reinforced plastic product released from the WPC mould; b) the WPC based mould for making boat parts printed at Centria UAS

Environmental concerns in Europe concentrate on restricting the use of finite resources and the necessary to manage waste disposal; as a result, the desire to recycle material in the end of their lives stages has increased (Yeh, Agarwal & Gupta 2009). WPC is environmentally friendly material because it is product of converting by-product or waste into new material. It plays a key role to reduce raw materials. It can also be recycled and renewed to be used again. Despite that, recycling of WPC mould also needs to be studied to contribute to circular economy and decrease pressure on environment.

3 WOOD PLASTIC COMPOSITES

Fast development of plastic relating to fossil fuel have a considerable consequence to the planet and environment. Recycling and reusing of plastic and other resources are one of the solutions for people to avoid exploiting raw materials and releasing waste to nature. Moreover, wood waste is also concerned that its end of life is only burned or disposed. Besides that, with non-stop development of industries, new materials with many advantages and wide applications are researched and developed. To take advantage of wood waste and plastic waste, wood plastic composite can be one of the promising alternatives to their raw materials (Winandy, Stark & Clemons 2004). Wood plastic composite is a moderately new material that have expanding market recently and numerous possible potentials (Tolinski 2015).

Wood-plastic composite is composed of wood fibres from saw dust or other cellulose-based fibre fillers such as pulp fibre and virgin or recycled polymeric material with small amount of additive. WPC has the best features combination between wood and plastic. The wood content of the material can range from 20% to more than 80% (Klyosov 2007).

3.1 Manufacturing

There are two main components using manufacture in WPC: wood fibres and thermoplastic matrix. Moreover, to improve their properties in many applications, additives with small amount are also added. Before product manufacturing, components of WPC are mixed. Then, the next step is compounding. Injection moulding and extrusion are two of the main manufacturing methods for WPC (Kim & Pal 2011). About extrusion, there are two different apparatus, a one-screw or a two-screw equipment. Most WPCs are manufactured to use in the construction industry. Additionally, compression moulding method is commonly used to manufacture WPC in the automotive industry (Carus, Eder, Dammer, Korte, Scholz, Essel, Breitmayer & Barth 2015).

Depending on the area of manufacture, both softwoods and hardwoods in the form of fibres, particles, or fine flour are raw material of manufacturing process. Fibre bundles or fine flour cell wall fragment are wood particles with an aspect ratio (the length to diameter ratio) of 1:1 to 5:1. The physical and mechanical properties of the WPC are affected by the structure of wood component (Clemons 2008). For example, tensile strength increases when aspect ratio of fibres is higher (Klyosov 2007).

In WPCs, the most popularly used thermoplastic matrix material are polyvinyl chloride (PVC), polyethylene (PE), acrylonitrile butadiene styrene (ABS), polystyrene (PS) and polypropylene (PP) (Oksman & Bengtsson 2007). The most preferred virgin plastic is polypropylene (PP), while polyethylene (PE) is used as recycled plastic in production (Ashori 2008).

Additives which are depended on target area of application can be colorants, coupling agents, stabilizers, reinforcing agent, foaming agents and lubricants (Kim & Pal 2011). The physical and mechanical properties of WPC mainly are depended on the interaction between wood and plastic. Although there are small amount of additives, it is very important in the improvement of the interaction (Wechsler, Hiziroglu & Ballerini 2009). There are two main groups of additives added into WPC, including mineral additives and coupling agents. Coupling agents help to increase tensile strength, while mineral additives can improve flame retardancy. The common additives are maleic anhydride-grafted polypropylene (MAPP) and maleic anhydride-grafted polyethylene (MAPE) for coupling agent, and calcium carbonate for mineral additive. Usually the polymer blend PE/MAPE and PP/MAPP were investigated as a benchmark for WPC (Yuan, Wu, Gotama & Bateman 2008). The additive is typically added with small percentages, less than 5% (Klyosov 2007).

Besides virgin material, different kinds of waste materials or by-products and recycled materials are possible to be used as raw materials in WPC production (Kim & Pal 2011). There are many studies related to different recycled materials in WPC. For instance, fibres from rice straw (Tawfik, Eskander & Nawwar 2017) and waste paper (López, Boufi, El Mansouri, Mutjé & Vilaseca 2012) have been used. Moreover, plastic waste such as waste from automotive industry (Cholake, Rajarao, Henderson, Rajagopal & Sahajwalla 2017) and electrical and electronic equipment (Sommerhuber, Wenker, Rüter & Krause 2017) have also been used.

3.2 Mechanical properties of WPC

Mechanical properties play an important role in choosing suitable WPC for different applications. Not only WPC can be manufactured in many colours, shapes and sizes, but also formed into any shape depending on the processing method (Taylor, Yadama, Englund, Harper & Kim 2009). With the development of catalyst technology and process engineering, WPC is also chosen more for wide application. WPC has properties of both the main ingredient: wood components and polymer (Sain & Pervaiz 2008).

Properties of WPC are quite flexible between different wood species, wood particle size characteristics and types of wood fibre (Stark & Berger 1997). Moreover, the interaction developed between wood and the thermoplastic material, which can be improved by using coupling agent as additive, is one of main factors affecting most of the physical and mechanical properties WPC (Lu, Wu & Jr 2000). Although the interphase and adhesion between the fibre and the matrix affect slightly on the composite stiffness, it can decide the strength, toughness and long-term properties such as creep and moisture stability. Chemical groups such as silanes on the wood fibre surface and the compatible chemical group in matrix polymer can create covalent bonding. Thus, while maleated polyolefins create a chemical bond on the fibre surface, the anhydride grafted polymer molecules entangle between the matrix polymer physically (Migneault, Koubaa, Perré & Riedl 2015). Therefore, the hydrophilic wood and hydrophobic plastic can be compatible to form single-phase composite by the additives (Wechsler et al. 2009). FIGURE 2 illustrates the comparison between some properties of WPC and ideal material proving WPC potential application. WPC can be seen as a promising material for outdoor application.

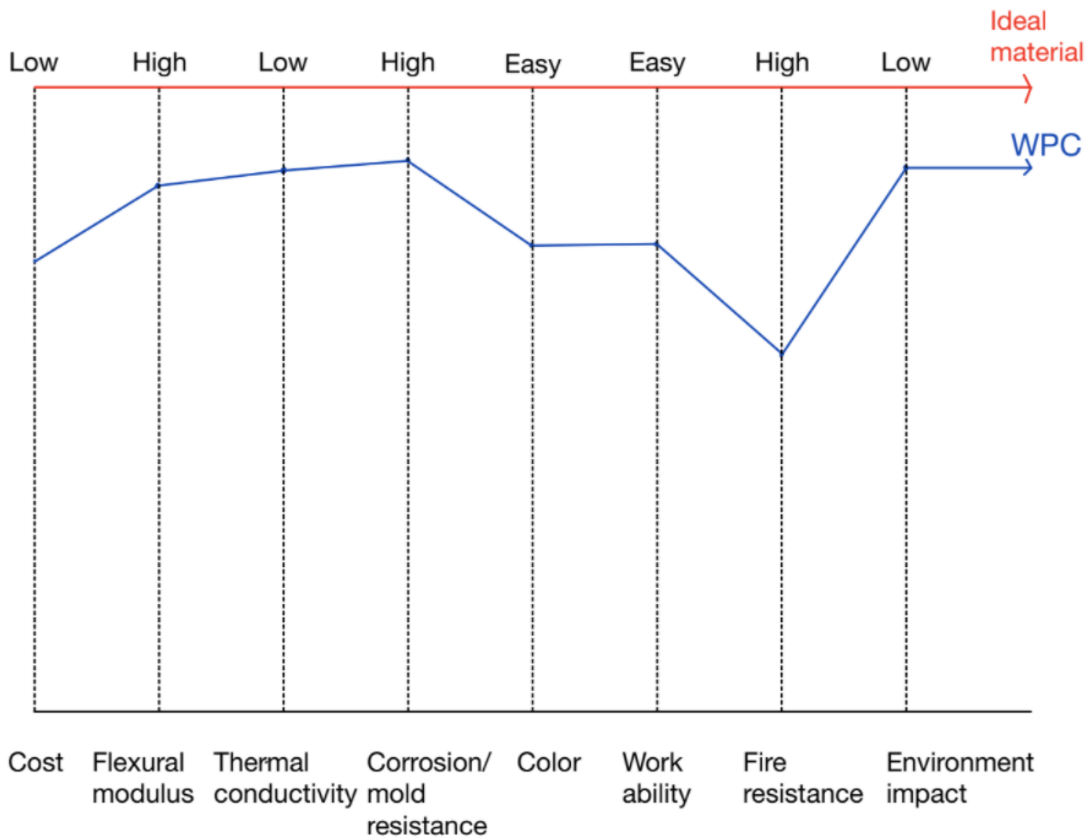


FIGURE 2 WPC properties compare to ideal material (adapted from Sain & Pervaiz 2008)

There are several improvements of mechanical properties of WPC compared to wood or unfilled plastic. Wood is added to a thermoplastic matrix to increase the mechanical properties and thermal stability compared to the solid thermoplastics, while thermoplastic component is moisture barriers to wood element, which helps WPC have water adsorption, swelling less than wood and traditional wood composites (Wolcott & Englund 1999). Although wood is the most common and inexpensive source of fibre available, it contains hydroxyl groups and other oxygen-containing groups that promote water absorption through hydrogen bonds. The WPC wood particles are completely covered with plastic, which allows moisture to penetrate only the exposed parts of the wood fibres and not through the plastic boundaries, making the composite product highly resistant to leaching. Thus, there is almost no humidity, risk of swelling and fungal attack (Sain & Pervaiz 2008). This property makes WPC suitable for outdoor applications where untreated timber products are not. Tensile strength and stiffness properties of WPC lies between properties of polymer and wood at acceptable level. Polyethylene and polypropylene-based WPC material is flammable. However, for some specific application, a low flammable WPC can be made by loading a WPC formulation with flame retardant components. (Klyosov 2007)

Waste wood and recycled plastic material can be used to produce WPC, therefore, WPC has low maintenance cost compared to only plastic or solid wood. Contributing on its economic benefits, WPC has low density and low friction during compounding, thus, there is almost no affect to equipment and environment (Kim & Pal 2011). Additionally, the recycling potential of WPC is promising in future. (Hiziroglu 2016)

Despite many benefits of this material, one of predominant drawback for production of WPC is low acceptant processing temperature, due to lignocellulose degradation that could impact on composite performance. Thus, the processing temperatures usually are kept above 200°C; however, it is possible to use higher temperatures for short periods. This condition restricts also the type of thermoplastics that can be used which are polyethylene (PE), polypropylene (PP), polyvinyl chloride (PVC) and polystyrene (PS) (Rowell, Field & Jacobson 1998). It is important to enhance the benefits of the composite and bring their properties to suitable applications by understanding the limitation and advantage of the composite.

3.3 Application of WPC

Good mechanical properties of WPC can be applied in many products with more flexible price and maintenance. Currently, WPC is mainly used for outdoor products. The most popular application of

WPC material is in building and construction, for example, outdoor deck floor, railings, fences, siding, window and door frames, panels and indoor furniture (Winandy et al. 2004). Some products using WPC can be resistant to ultra-violet light, colour fading and rigorous weather conditions such as rain, snow or scorching weather (Matuana & Stark 2015). Moreover, WPC also participates in other sectors, such as industry and consumer products, automotive interiors and household use (Biron 2017). FIGURE 3 is a WPC decking product of UPM company in recent market.

FIGURE 3 Wood plastic composites are used mainly for producing outdoor products such as UPM Formi decking (adapted from UPM ProFi Deck)

One of their applications is WPC mould. WPC material can almost replace the thermoset in mould. However, coating surface layer is still thermoset to ensure working function of the mould. This reduces high amount of thermoset material using in industry (Huhtanen, Rajan & Rainosalo 2021).

3.4 Recycling process of used WPC

Although WPC is a "green" material, the recycling and reuse of materials also show more opportunities for producing new products with comparable properties. While the WPC has high attention because of using recycled plastics for production, the WPC recycling is still missing (Krause et al. 2018). By studying the properties of recycled WPC, the reprocessing of WPC can be modified to its mechanical properties. From that, circular economy approach can be established for WPC sustainable development.

Many studies have been investigated on recycling of different types of WPC with different mechanical processes. Though mechanical processes, there are changes in physical and mechanical properties of WPC. For example, wood fibre/polypropylene composites indicated a decrease of 21% in tensile strength and 17% in the tensile modulus after going through six extrusion cycles (Dickson, Even, Warnes & Fernyhough 2014).

Composites consisting of wood fibre (60 wt.%) and high density polyethylene (HDPE) (40 wt.%) were reprocessed in twin screw extrude, which presented mechanical properties that were reduced after every single recycling cycle due to the low adhesion between the HDPE and wood flour (Shahi, Behravesh, Daryabari and Lotfi, 2012). Another example is that tensile strength of cellulose fibre/polypropylene (PP) composites was reduced by 25% and tensile modulus by 16% after up to eight twin screw extruder cycles (Beg & Pickering 2008). Thus, several limitations have to be considered, such as thermal degradation, the decrease of wood fibre length and breakage of polymer chain (Krause et al. 2018); (Hietala, Niinimäki and Niska, 2011). The decrease in mechanical properties is also caused by changing the interfacial bonding between the polymeric matric and wood fibres (Shahi et al. 2012).

Over the recycling times, the mechanical properties such as tensile strength, thermal degradation, fibre length are usually reduced; however, in some researches, the stiffness can also be improved due to the increased dispersion of the fibre within the polymer matrix (Dickson et al. 2014); (Beg & Pickering 2008).

3.5 Fused Granular Fabrication method for 3D printed WPC products

WPC pellets can be used to feed into Fused Granular Fabrication (FGF) 3D printer to create 3D printing products. FGF is one of methods used in the 3D model which is printed by layers. FGF is also known as pellet extrusion printing, pellet printing, fused pellet fabrication or fused particle fabrication, because the FGF uses granules or pellets as feedstock. Pellets are fed into a feed section, where they go through a screw. Material is pushed by the screw to the extruder and into the heated part. Then, the melted material is forced to flow through a nozzle, which can be customized to fit with print speeds or finer details. After printing, the part is placed to be cooled and solidified to get the final product (Stopka, Kohar, Weis and Šteininger, 2018). FIGURE 4 illustrates the main parts of FGF printing system.

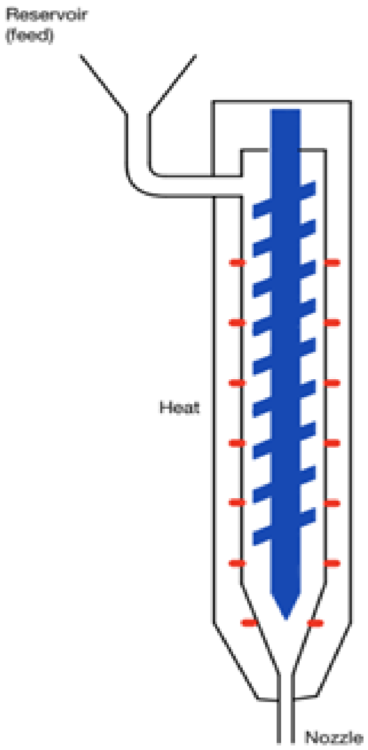


FIGURE 4 Fused Granular Fabrication printing system (adapted from Stopka et al. 2018)

Different from the Fused Filament Fabrication (FFF) technique, the FGF can print from pellet material, that eliminates the process of filament manufacturing. Therefore, FGF leads to save more cost and time, and simplify operation in printing process (Stopka et al. 2018). Moreover, Fused Granular Fabrication (FGF) has high potential to play an important role on recycling plastic waste (Woern, Byard, Oakley, Fiedler, Snabes & Pearce 2018). According to Green Fablab project, a number of virgin polymers and post consumers waste were recycled successfully by the FGF systems (Byard, Woern, Oakley, Fiedler, Snabes & Pearce 2019). Researches about the printability for recycled polymers with FGF can lead to new recycling loops of polymers (Cruz Sanchez, Boudaoud, Camargo & Pearce 2020).

4 EXPERIMENT

The recycling research occurred with two processes. Firstly, products as recycled moulds were made. Then, the products including two phases, powder and plates were mechanically analysed for studying the properties changes. The experiment included five cycles in recycling waste of wood plastic composite mould with waste powder input by compression moulding method. Plates are products after compression moulding, which represent mould products.

The detail of whole recycling process with compression moulding is shown in FIGURE 5. While powders were used for Melt Flow Index testing, plates were used for density, tensile test and optical microscopy. For tensile testing, the plates were prepared and cut to become test specimens for further testing. There are five cycles of recycling process from powder to become plates as products. Each stage of recycling process is described further.

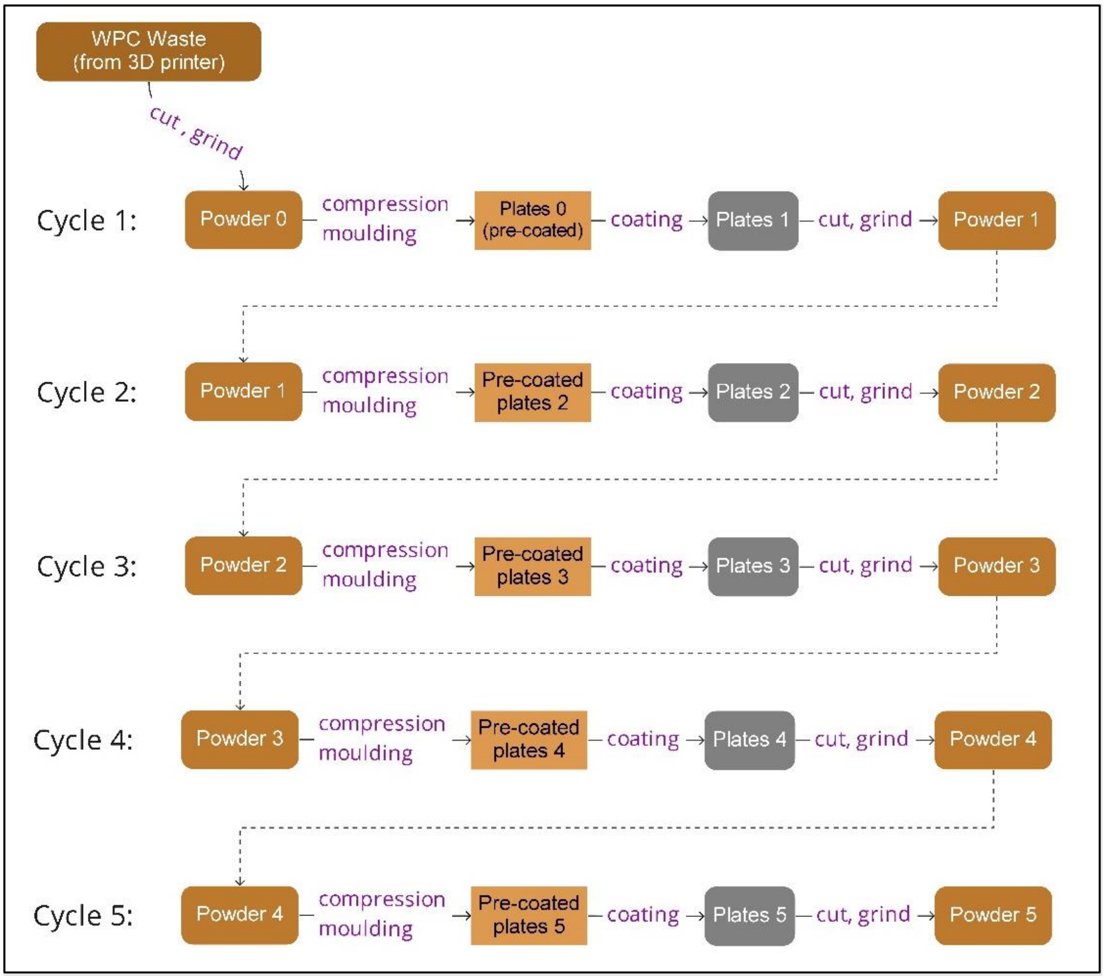


FIGURE 5 Detail recycling process with five cycles from 3D-printed WPC waste

4.1 Raw material

As explained above, the raw material for this study was waste generated from 3D printing. The printing was done by fused granular fabrication (FGF) (FIGURE 6 a) of wood plastic composite (WPC) granules containing 40 wt.% of wood fibres at 215°C. The WPC was produced by melt mixing 57% polypropyl-ene, 40% wood flour and 3% MAPP in a co-rotating twin screw extruder. During the printing process, the test models of the mould and rest of the waste generated were collected which acts as raw material for this study (FIGURE 6 b). Besides that, the reference plate was made from wood plastic composite granular by compression moulding method.

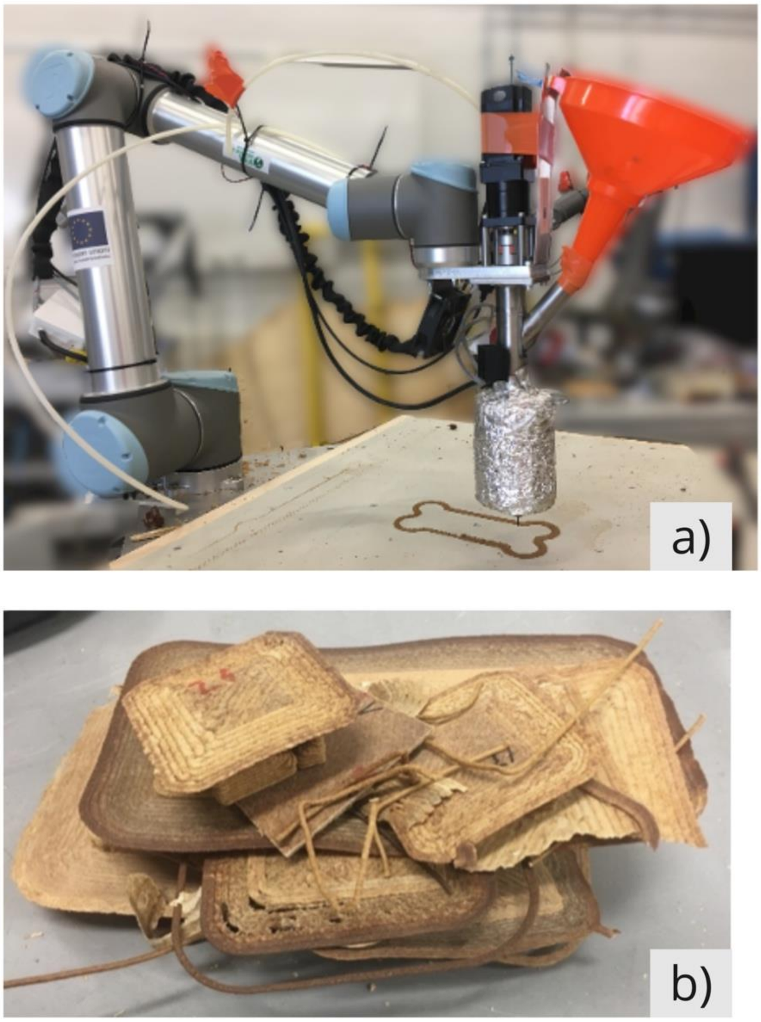


FIGURE 6 a) Fused Granular Fabrication 3D-printer and b) WPC waste made by FGF 3D-printer

The main chemical used in the experiment was a black coloured MAXGUARD GT // HF / SF premium tooling gelcoat and hardener. Tooling gelcoat is based on epoxy vinyl ester resin. Hardener is Methyl

Ethyl Ketone Peroxide (MEKP), used as catalyst for initiates the crosslinking of unsaturated polyester resins.

4.2 Procedure in detail

Recycling stage firstly started with size reduction by band saw and grinder. In the next step, the powder was compression moulded by hot press machine. Then, the tooling gelcoat layer was applied on one surface of each plate. From that, powder and plates were created to further research. Recycling process in FIGURE 5 will be described in detail, including cutting, grinding, compression moulding and plate coating process.

4.2.1 Cutting and grinding

Before going to compression moulding, the 3D-printed waste or plates were grinded to be powder. Firstly, the WPC waste from FGF printing were collected and cut into smaller pieces by a band saw (FIGURE 7 a). The machine ran with 11 m/s cutting speed and 2.5 amps motor. The size of pieces had to be smaller than 4cm x 4cm x 4cm based on the feeder of the grinder to prevent faults while grinder operating and uneven size of output powder after grinding. FIGURE 7 b) shows examples of pieces after cutting.

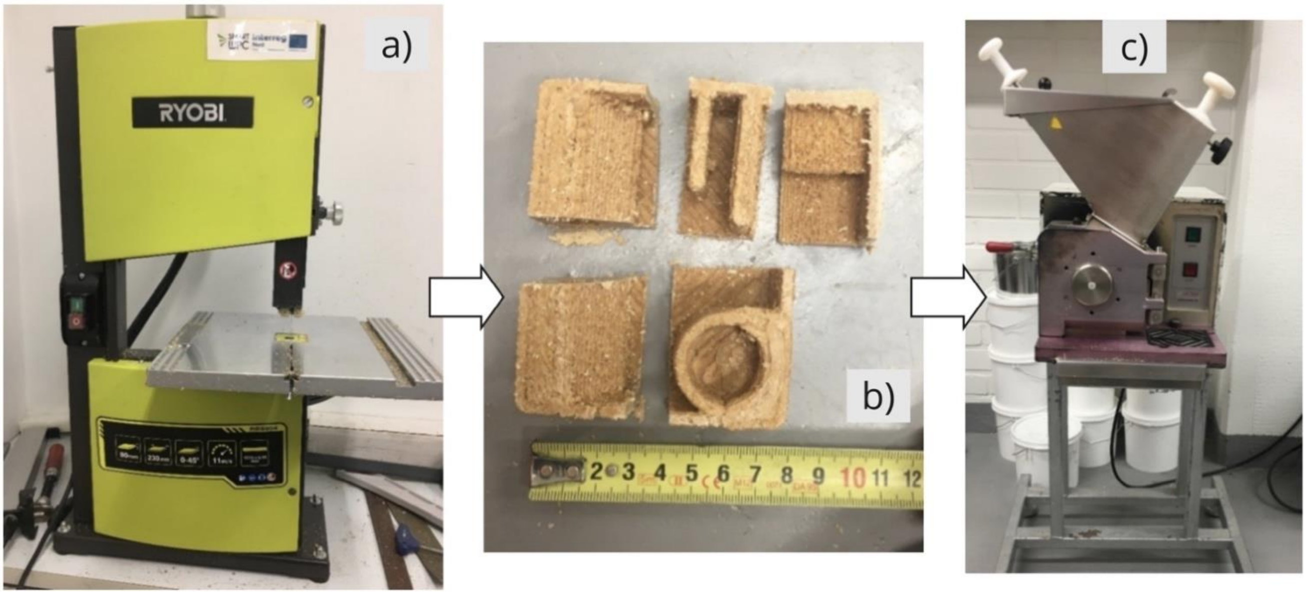


FIGURE 7 a) Used band saw in cutting process, b) Cut 3D-printed waste samples and c) Grinder machine used in the grinding process

After the cutting process, those pieces were transformed to grinding machine to make the powder of WPC waste. Grinding machine was used with 6 mm square perforation (FIGURE 7 c). For other next cycles, input source were the plates as products of the cycle before. The lost material was average 13.5 g in cutting process and 17 g in grinding process.

After the grinding process, powder was collected and used to create plates by compression moulding technique. In FIGURE 8, the change in colour between powder from different cycles can be seen. The colour change in grinded wood powder is occurring due to the continuous exposure to the thermal and mechanical processing. The increase in tooling gelcoat content after each cycle can also be observed. These effects made the colour of powder became darker when going through every recycling process.

FIGURE 8 The colour changing of powder of each recycling cycle after grinding

4.2.2 Compression moulding

Compression moulding is one of techniques used in composite manufacturing processing. The feeding material is placed in a mould, heated and close-shaped by a hydraulic press (Edebali 2021). The material including thermoplastic is in granular form. In this case, while reference plate was made from wood plastic composite granular, plates of other recycling process were made from waste powder which was produced from the previous steps. The material was placed in a hot mould. In this experiment, the mould size was 20 cm x 20 cm. Amount of material was 150 g per one plate. The operation temperature was 190-200℃ with setting at 200℃. The mould was closed by a hydraulic press at 12 metric tons in 2 minutes. Next, the whole system was cooled down with cooling air and water flow to room temperature. During the cooling time with cooling air and water flow, the press force was maintained at 12 metric tons. After that, the mould was opened, and the plate was taken out. Normally, the lost material was around from 1 to 4 grams per plate. FIGURE 9 illustrates those plates from different recycling cycles.

Because amount of the tooling gelcoat increased in every next cycle, the colour of plates also has same darker trend as colour of powder.

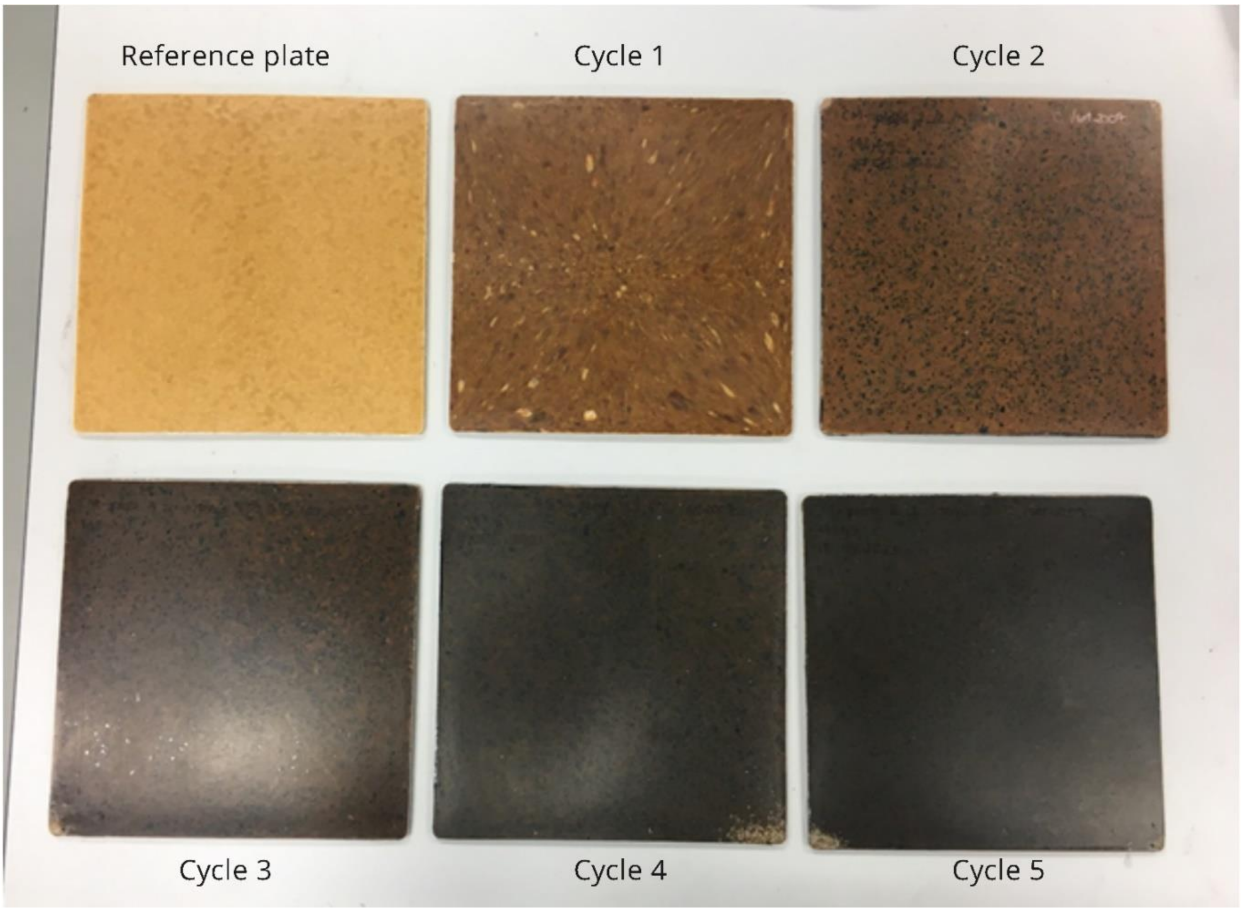


FIGURE 9 Output plates of each recycling cycle after compression moulding

4.2.3 Plate coating

After compression moulding, the plates were applied a layer of tooling gel on one surface. The coating step is important because the tooling gelcoat helps the mould be resistant to heat, improve mould quality, increase lifetime and reduce cleaning effort. FIGURE 10 a) shows used tooling gelcoat (Epoxy vinyl ester resin) and hardener MEKP. The used tooling gelcoat was black MAXGUARD GT Premium Tooling Gelcoats (Ashland 2017). MEKP is used as a hardening agent that speeds up the curing reaction of polyester resin and vinyl ester with temperature. The chemical reaction in resin with the styrene monomer are taken place to allow crosslinks to form between monomers. The components in the liquid resin are connected by theses crosslinks. The resin turns from liquid to solid phase, when most of the cross-links have formed (Toorkey, Rajanna & Prakash 1996).

Black epoxy vinyl ester resin base tooling gelcoat was taken in the needed amount. MEKP was added to the tooling gelcoat with 2% weight of gelcoat. The mixture was mixed well to ensure all the gelcoat was catalysed. A brush grade gelcoat was applied carefully on a surface of each plate to get even layers without air bubbles. Then, coated plates were transfered into chamber at 60℃ to be cured for 24 hours. For example, the coated surface is shown in FIGURE 10 b).

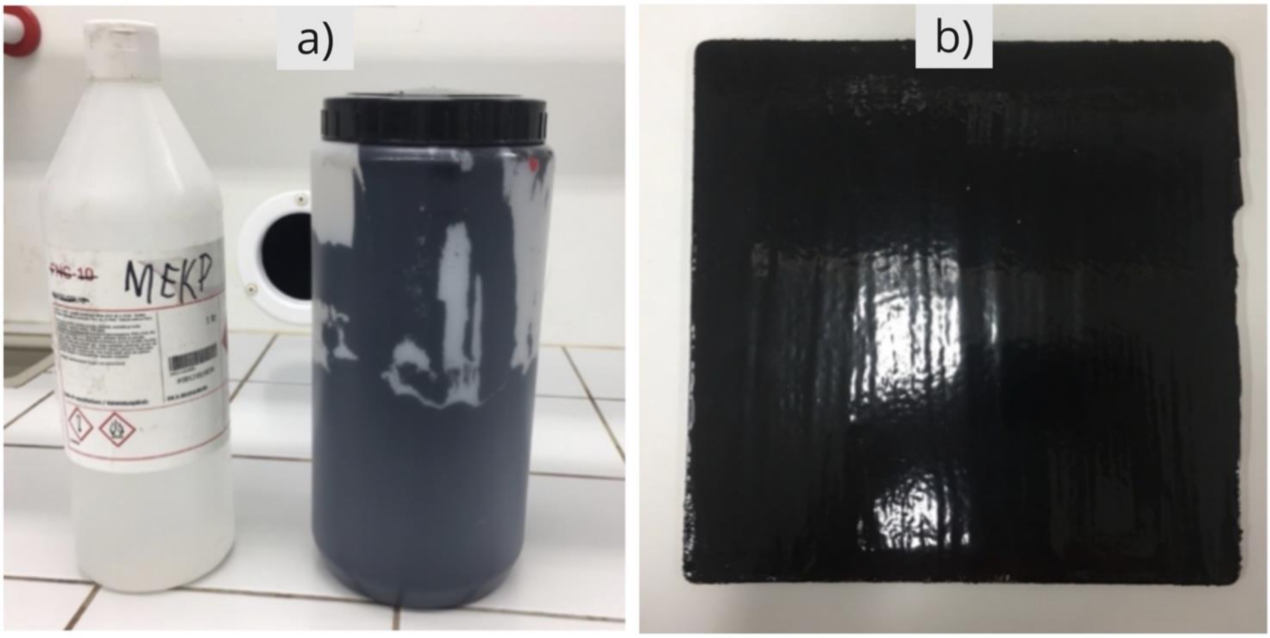


FIGURE 10 a) Tooling gelcoat and hardener MEKP using in plate coating process b) Coated surface of plates after curing

5 TESTING METHODS

In order to study mechanical properties of recycled WPC moulds and the change of their properties through every recycling process, the powder and selected plates of each cycle were tested. There were melt flow index test, density, tensile test, gelcoat particle size measurement and optical microscopy cross section at break point. FIGURE 11 shows detail of testing process and which type of products were tested.

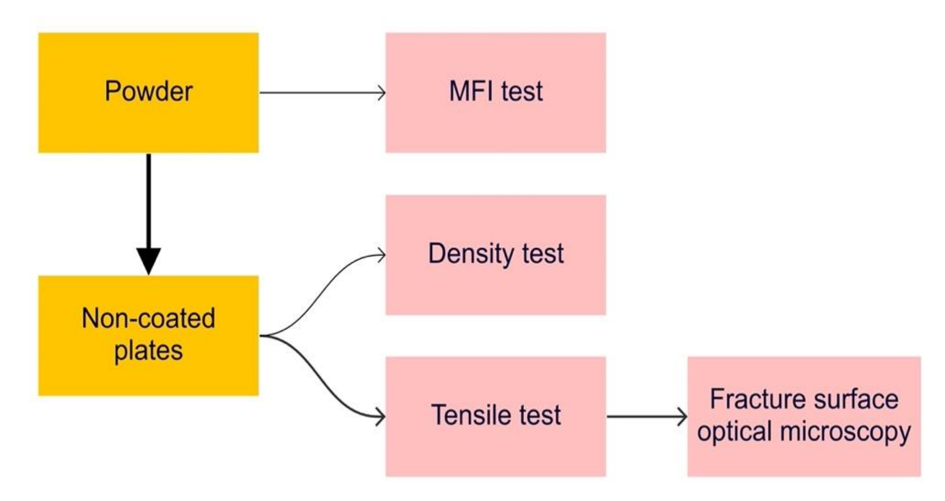


FIGURE 11 Testing method for WPC powder after grinding and non- coated recycled WPC plates after compression moulding

5.1 Melt Flow Index (MFI)

The melt flow index (MFI), which is a measure for flow properties of melted plastics, is used to thermo-plastic quality control in the plastic industry. The melt flow index test is one of the most popular method to measure material viscosities. Melt flow index shows the output flow rate in grams that occurs in 10 minutes through a standard die. A fixed pressure is applied to the melt via a piston at a fixed temperature. The flow has higher MFI meaning the more polymer flows under test conditions.

The MFI measurement was implemented following EN ISO 1133 (ISO 2005). The test parameters were the temperature at 210°C, time interval in 30 seconds, cutting 10 times and the press load by 10 kilo-grams. The test material was measured by about 5 grams per test. When the set temperature had reached, the material was added into the cylinder by a funnel and a charging tool to remove entrapped air. After the basic weight was added, the whole system was allowed to heat up in 4 minutes. Next, additional weights were placed on the basic to get 10 kilograms for the total weight. Before starting the test, the flow of material needed to be without air bubble. There were 10 cutting pieces of material released from out flow material. Those pieces were weighed to get the average weight of one piece or how much material went out in 30 seconds. From that, the MFI could be calculated by multiplied 20 times for material went out in 10 minutes. To sum up, the MFI can be calculated by EQUATION 1.

The MFI, expressed in grams per 10 min, is given by the formula

$$\text{MFI}(T, m_{nom}) = \frac{600\,m}{t} \qquad (1)$$

where T is the test temperature, in degrees Celsius; m_{nom} is the nominal load, in kilograms (10 kg in this research); m is the average mass, in grams, of cut-offs; t is the cut-off time-interval, in seconds (30s in this research); 600 is the factor used to convert grams per second into grams per 10 in (600s).

5.2 Density

Density of WPC made from pellets and from grinded recycling process was measured. From non-coated plates of each cycle, pieces which had 2 cm x 1.5 cm dimension size were cut for measuring density. The method for density determination applied Archimedes' principle. The density of solid can be found with the use of a liquid at known density and weight of solid in air and in the liquid (Clark 1962).

Density measurement applied Archimedes' principle

$$\rho = \frac{A}{A-B}\rho_0 \qquad (2)$$

where ρ is solid density, ρ_0 is known liquid density, A is solid weight in air, B is solid immersion weight.

5.3 Tensile test

Tensile testing is one of most fundamental and common mechanical tests. During the test, a tensile force is applied to a specimen and determine its response to the force. The test will end when the specimen is broken. The purpose of tensile test is to control material quality and in material development research.

In this experiment, the tensile testing was done according to standard ISO 527-2:2012, using Tiratest 2705 testing machine equipped with a 5 kN load cell. The initial distance between grips was 115 mm. The test rate was 2 mm/min. The test specimens were cut and prepared from a selected coated plate of each cycle. However, due to weak adhesion between the coating and substrate, most of the coating was removed during the cutting procedure. Therefore, the remaining coating was also removed from the test specimen and test was done with specimens without any coating remaining on the surface. FIGURE 12 is an example of a test specimen. A plate could be made to 6 test specimens.

FIGURE 12 Test specimen that was cut from non-coated plate for tensile testing

5.4 Fracture surface optical microscopy

From tensile test, all test specimens were observed under an optical microscope with magnification x1.5. The area for observation was at break cross section after tensile test. The purpose is to know how coating particles size and distribution is affected by repeated recycling and to analyse the adhesion between polymer, fibre and the coating particles.

6 RESULTS AND DISCUSSION

In this chapter, the mechanical properties including MFI, density, tensile strength and optical microscope of recycled products are illustrated by the data, photos and graphs. The research results show variation of properties through recycling process, which can contribute on predicting the further recycling stages. The reasons of the variation are also discussed in this chapter.

6.1 Melt Flow Index

Each powder of a cycle was tested five times with 50 samples. The weight of output material was determined as gram per 30 seconds. Then, the MFI was calculated as output material gram per 10 minutes by multiplied with 20. From average results of MFI, the bar graph in FIGURE 13 describes the increase trend of MFI through recycling process.

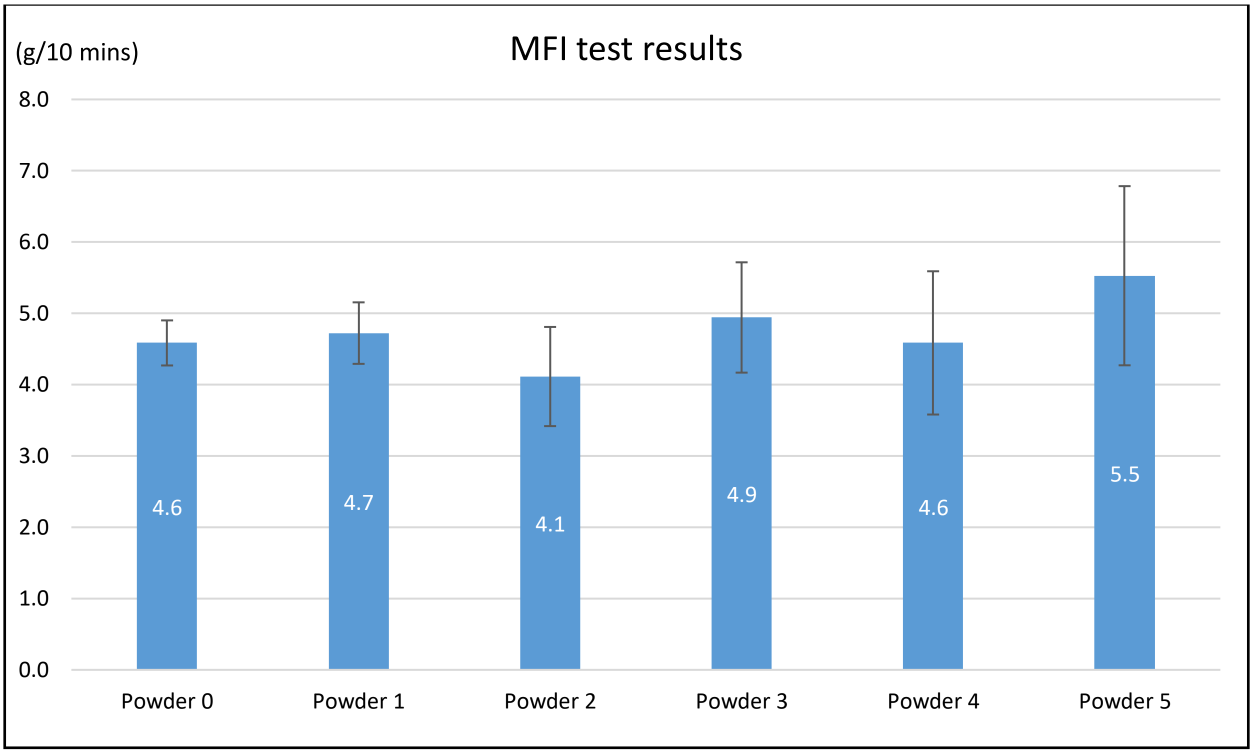


FIGURE 13 MFI test result graph of powder on each recycling cycle

During the testing, the coating particles are small enough to keep the material come smoothly out of the die of MFI without any blocking. The MFI of powder 2 is lowest at average 4.1 g/10 mins. While the powder 4 has the same MFI result to the powder 0, the MFI of powder 5 is the highest at average 5.5 g/10 mins. The MFI of powder 5 increases 19.5% comparing to powder 0. A higher MFI indicates a lower material viscosity; therefore, the material viscosity reduces through each recycling cycle. The lower viscosity shows the easier draining through the nozzle of the 3D-printer or the die of MFI (Bridges & Robinson 2020).

The explanation for the slight increasing melt flow of PP-based composites is the decreasing viscosity of neat PP with repeated recycling due to some thermo-oxidative degradation (Balatinecz & Sain 1998). In addition, the increase in MFI values of powder 5 compared to powder 0 is assumed to be also affected by the increase of coating particles content during every recycling cycle.

6.2 Density result

Average density of test sample from reference plate and non-coated plates of each recycling cycle was determined. From the result, the bar graph in FIGURE 14 describes the comparison density between the products of different recycling cycle and reference plates. There is a rising trend in density through every recycling cycle.

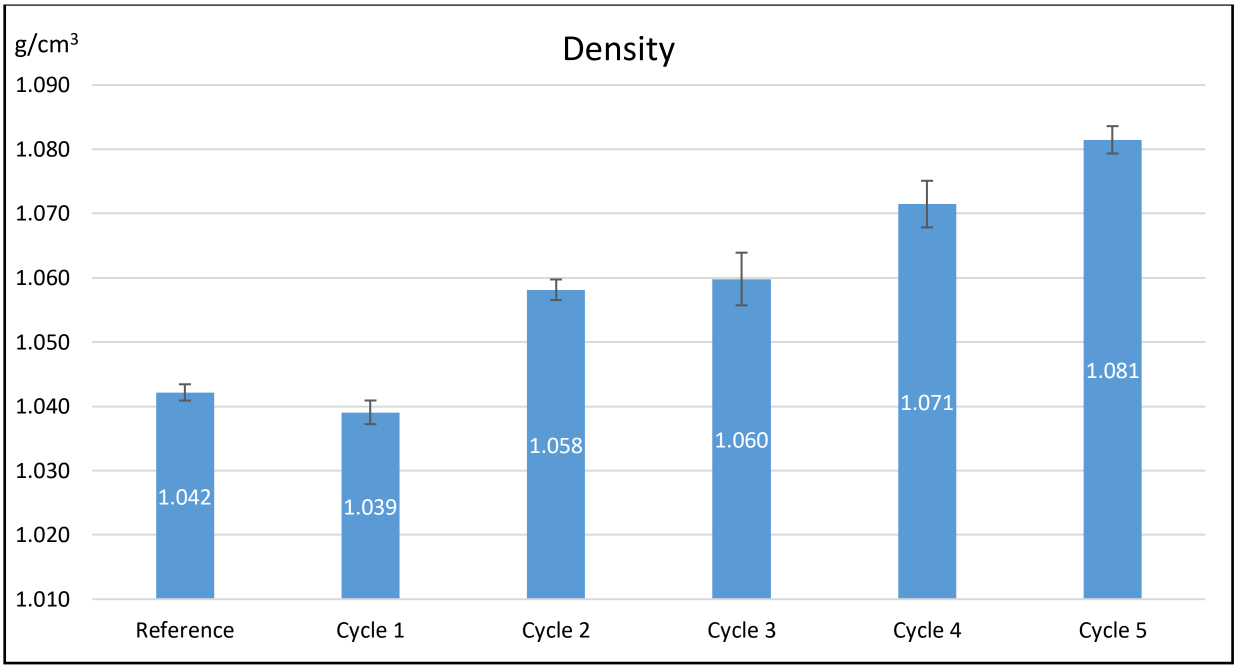


FIGURE 14 Density of non-coated plates by recycling cycles comparing to reference plates

While average density of plates in recycling cycle 1 (1.039 g/cm^3) reduces slightly comparing to reference plate (1.042 g/cm^3), the increase can be seen clearly from the result of recycling cycle 2 (1.058 g/cm^3). Comparing to the reference plates, the average density of plates in cycle 5 grows by 3.7%. According to Shahi, when recycling WPC without coating particles, the reduction in density was observed (Shahi et al. 2012). However, the results from this study show that the density increases with each recycling stage. Therefore, the increase in the density of composites can be attributed to the increasing amount of tooling gelcoat particles with density around 1.2 g/cm^3.

6.3 Tensile properties

From the result in FIGURE 15, the average max tensile strength of test specimens of cycle 1 is highest (24.03 N/mm^2), it is even higher than the reference plate's result (20.56 N/mm^2). The cycle 2's test specimens could bear at 21.22 N/mm^2, at nearly same strength as reference plate. However, the results of cycle 3, 4 and 5 have lower strength compared to reference plate and cycle 1. The max tensile strength of cycle 5 composites (16.66 N/mm^2) decreased by 30.7% compared to cycle 1 or 19% compared to reference plate.

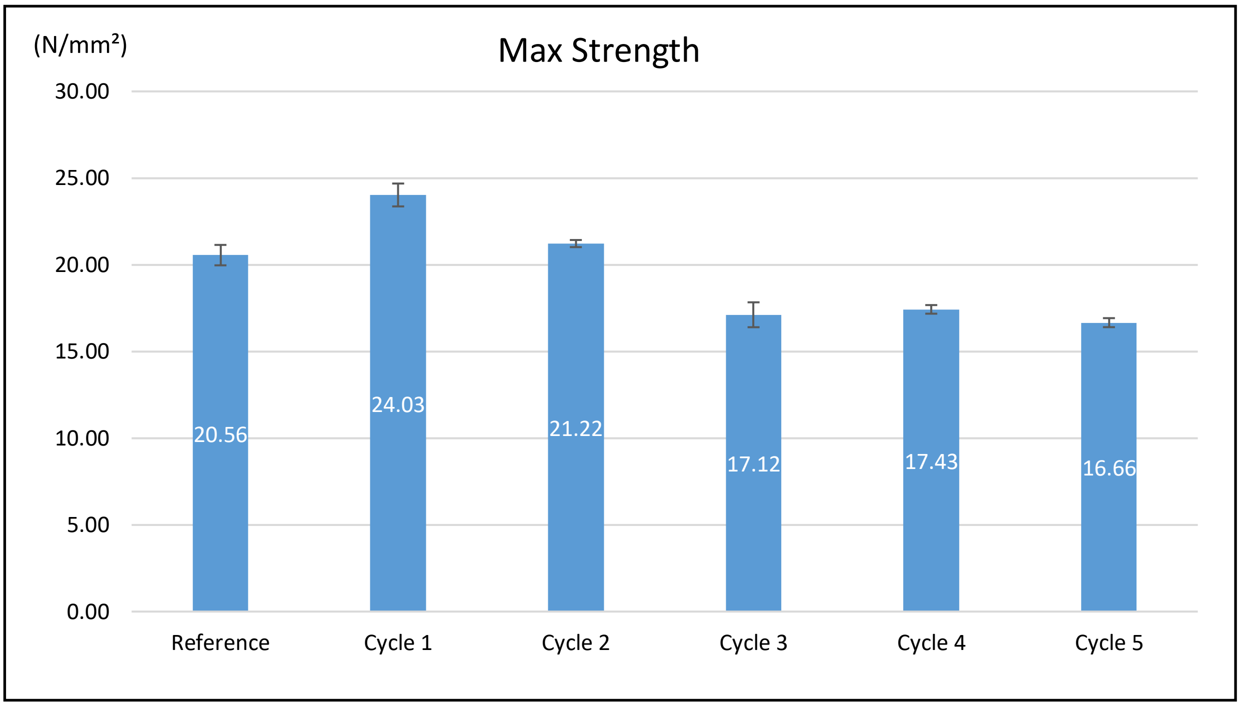


FIGURE 15 Average max tensile strength that test specimens of each recycling cycle went through during the tensile testing

The strain at break point in FIGURE 16 shows similar trend observed in the case of max the same trend. The highest percentage of strain is test specimens of cycle 1 at 9.02%. The result of reference plate and cycle 2 are nearly equal, respectively 6.76% and 6.45%. However, the strain of specimens of cycle 3, 4 and 5 has a decrease in strain at break, below 5%. The strain at break of test specimen cycle 5 is the lowest result, 3.72%.

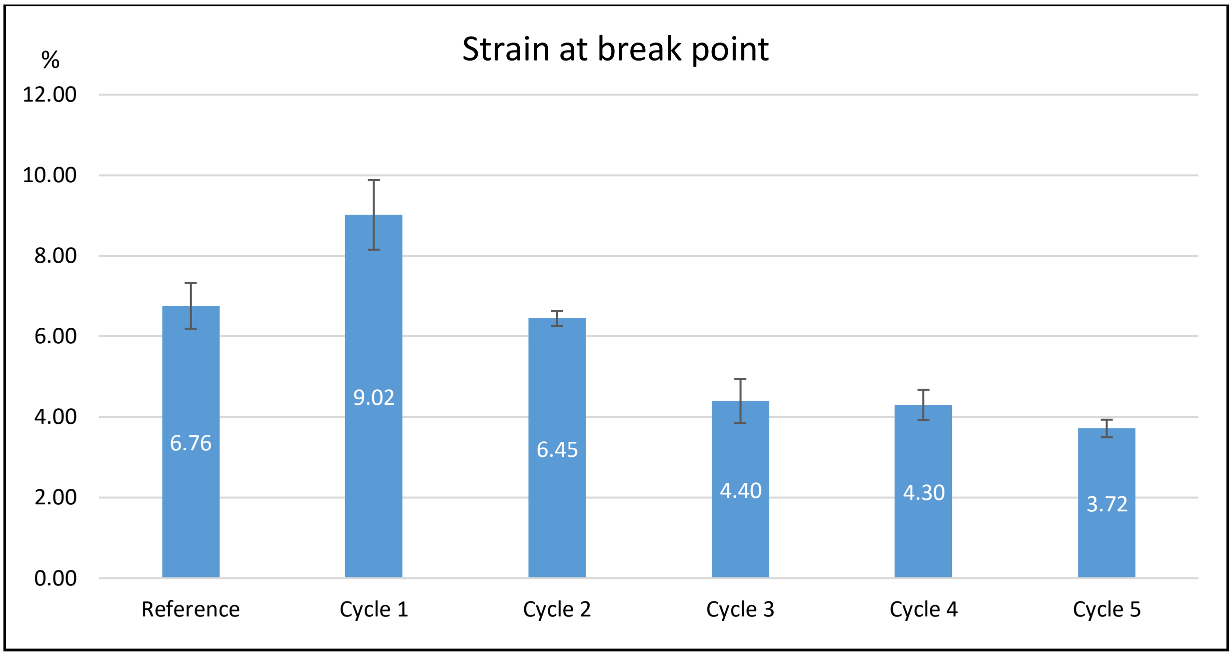
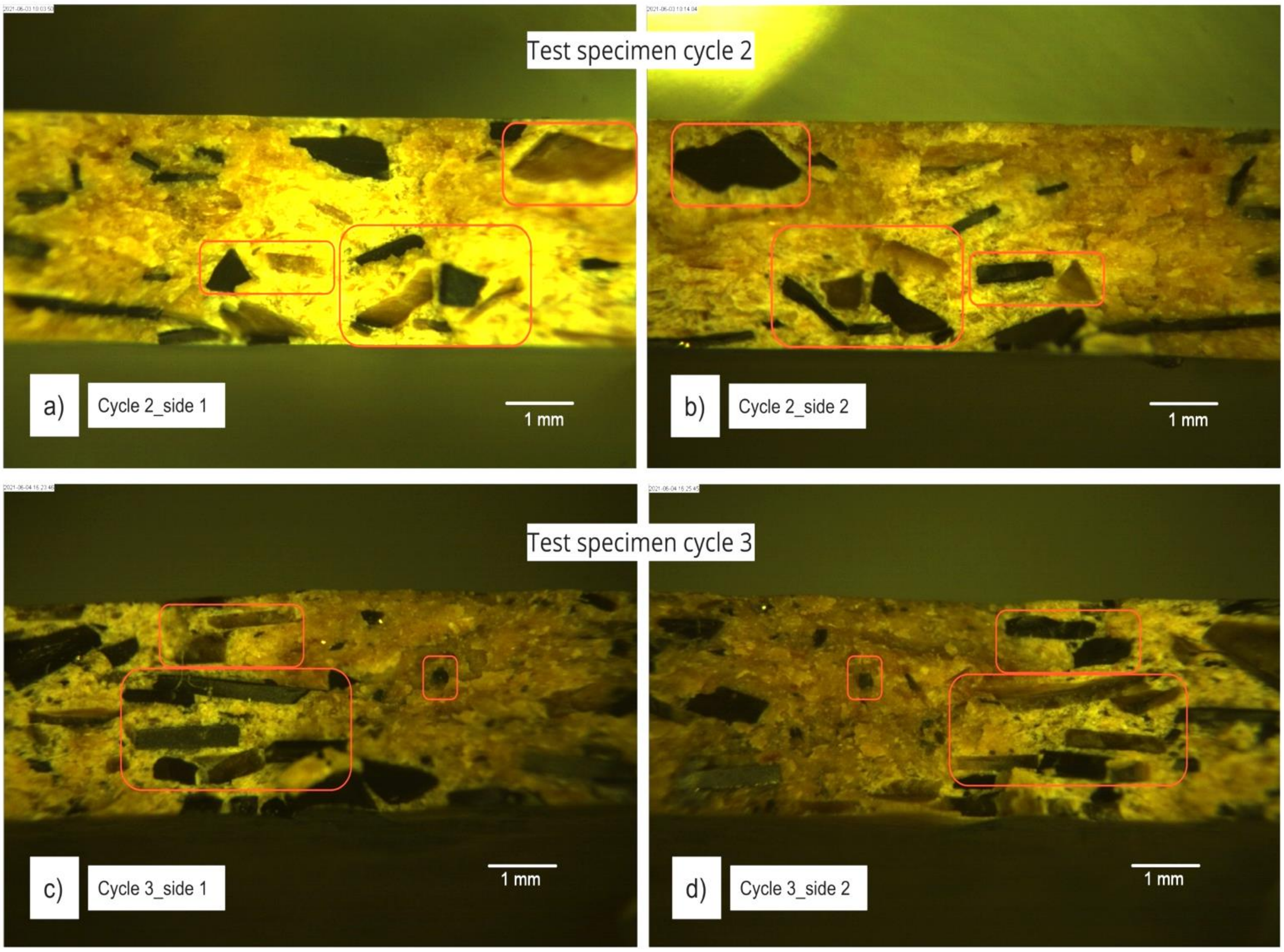


FIGURE 16 Average strain at break graph that test specimens of each recycling cycle went through during the tensile testing

Therefore, tensile properties can be improved and stayed stable through two recycling cycles. After that, tensile properties reduce, but they can stay stable at least three more recycling cycles. The increase of tensile strength in cycle 1 can be explained by the improvement of fibre dispersion. The dispersion ability can enhance because reduction of the fibre length and also decrease of the viscosity after each recycling process (Le Baillif & Oksman 2009). Nevertheless, the decrease of tensile properties from cycle 3 to cycle 5 is highly influenced by increasing tooling gelcoat particles content. The weaker adhesion between coating particles and WPC after each recycling process would be discussed further below.

6.4 Fracture surface optical microscopy

In order to study further about the change of tensile properties of recycled WPC with tooling gelcoat content and factors that impact the breakage in tensile testing, the fracture surfaces of test specimens were observed under a microscope. In FIGURE 17, photos from optical microscopy illustrates the break area of represent test specimen of each cycle when they went through tensile testing. All photos were taken at same magnification x1.5 to compare the size WPC and coating particles. With two pieces of a test specimen after breakage, two fracture surfaces of two sides are also compared to each other.



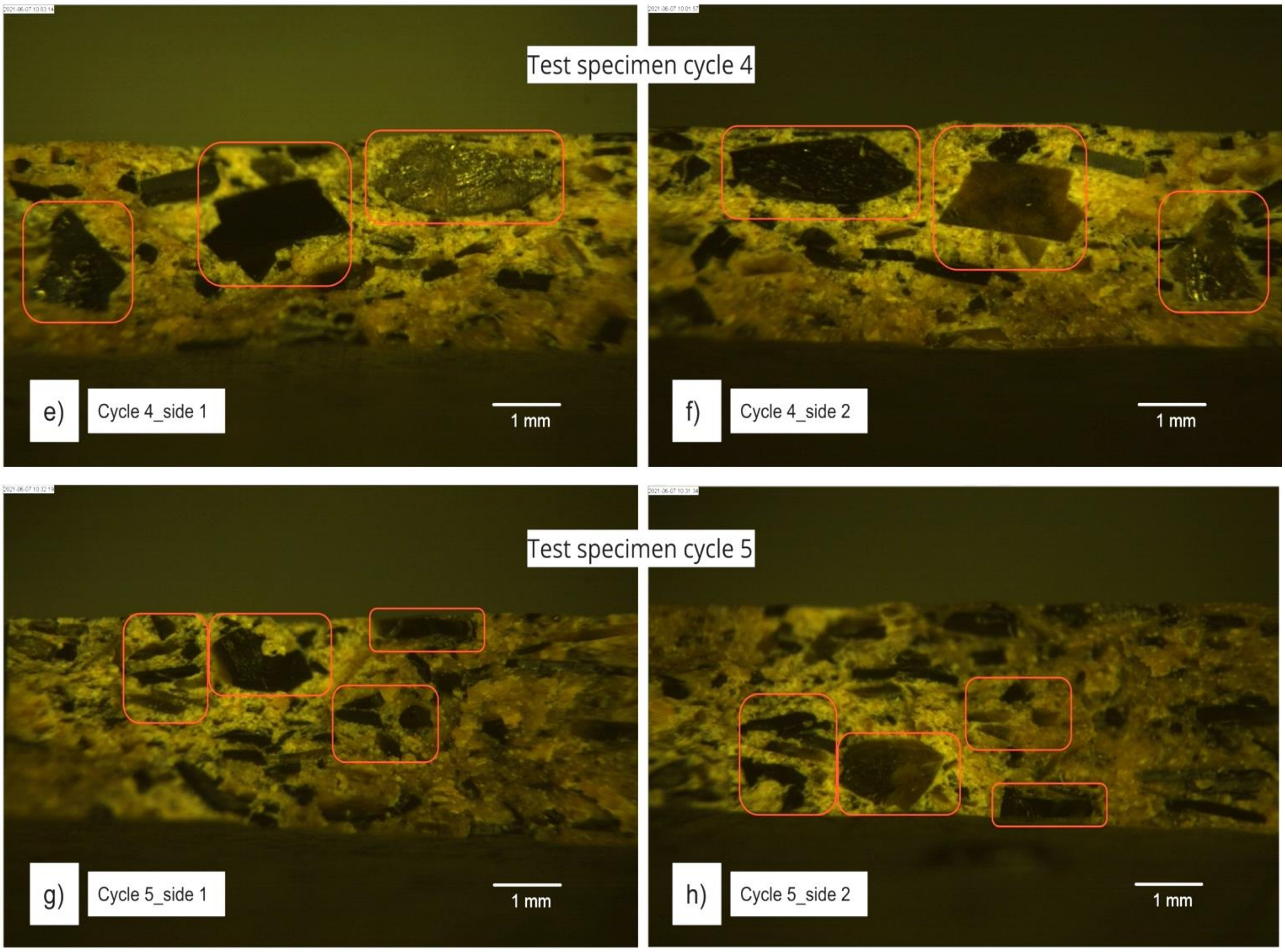


FIGURE 17 Optical microscopy in two sides fracture surfaces of represent test specimen for each recycling cycle

From recycling cycle 2 to cycle 5, the amount of coating particles content in these areas increases. Additionally, from cycle 2, test specimens have many small coating particles and some big coating particles. Small coating particles continued increasing through each recycling process. The tooling gelcoat particles included the old coating particles that were reprocessed to be smaller, and the new coating particles that were added to the material when new coating layers were applied on output plates in each recycling cycle. The large coating particles which can be recognized clearly are the new particles that were not processed through a complete recycling cycle. Moreover, this test can show the reduce of wood fibre length when the material was recycled.

In fracture surface of specimen cycle 2, there are coating particle areas that can be easy to compare two sides with their shapes. From the red highlight areas in microscope photos in FIGURE 17, it can be clearly seen that two fracture surfaces of two sides in same test specimens are symmetric in coating particles shapes. Therefore, the break of tensile testing can be affected by the weak adhesion between coating particles, wood fibres and polymers in that area. Tensile properties of WPC decrease from cycle 2, which can be also explained that the increase of coating particles content leading to larger areas that had weak adhesion between components of composite.

Besides that, the strength of composite materials is depended on not only interfacial adhesion, but also fibre orientation (Kan & Demirboga 2009). Therefore, with optical microscopy results, the decrease in tensile strength can also be influenced by the freely fibre orientation in this study. According to Gibson, oriented fibres in composite structure can perform better mechanical properties such as tensile strength than randomly oriented fibres (Gibson 2016).

7 CONCLUSION AND OUTLOOK

Wood Plastic Composite has been used for a long time with wide applications. WPC has many advantages, such as financial benefits, environmentally friendly material and good mechanical properties. Moreover, recycled components including recycled plastics and wood waste can be used for manufacturing the WPC. These days, WPC can also take part in mould production, because of requirement of thermoset replacement. WPC products such as mould have been designed and printed easily with 3D printing technique. However, the amount of WPC moulds can be large in future due to their development, which requires recycling of WPC moulds.

During five recycling cycles of WPC moulds by compression moulding, mechanical properties of their products were studied. There were melt flow index test, density, tensile test, and optical microscopy at fracture surfaces. MFI result of material powder shows a slight fluctuation and then in general increasing trend, which shows the easier draining through the nozzle of 3D-printer. Density of non-coated plates result illustrates nearly the same results of reference plate and cycle 1, but from recycling cycle 2, the density rises significantly because the coating particles content increase with recycling cycles. About tensile testing, going through two recycling cycles can keep their tensile properties due to improvement of fibre dispersion; however, the coating particles also cause the reduce of tensile properties from recycling cycle 3. Moreover, the break of test specimens through tensile test was studied further with optical microscopy. The symmetry of orientation of coating particles proves its effects on weaker adhesion between components of composite.

In conclusion, WPC with tooling gelcoat can go through at least two recycling cycles by compression moulding, which still maintains the mechanical properties of WPC as reference products. Depending on the application that recycled material participates, 3D-printed WPC can also be recycled more than two times with lower properties but might be still in acceptable levels. This research result opens more pathways for future research. For example, additives such as coupling agents can be added to improve the adhesion of composite components. According to Luo et al, dispersion and adhesion could continue improving with up to a MAPP concentration of 35wt% (Luo, Benson, Kit & Dever 2002). Furthermore, in order to recycle the 3D-printed WPC products with more life cycles, the coating particles removing technique should be considered.

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