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This project has received funding from the European Union's Horizon Europe research and innovation programme under the Grant Agreement No **101057765**

GREEN-LOOP

Sustainable manufacture systems towards novel bio-based materials

WP5 – Wood composites material production

D5.6 – Wood composite properties from tribological evaluation

Document information

Contractual Due date: 31.08.2024

Delivery Date: 31.08.2024

Author(s): FHF, IDE, NCC

Lead Beneficiary of Deliverable: FHF

Dissemination level: PU - Public

Nature of the Deliverable: Report

Internal Reviewers: FHF, IDE

GREEN-LOOP Key Facts

Project title	Sustainable manufacture systems towards novel bio-based materials
Starting date	09/01/2022
Duration in months	36
Call (part) identifier	TWIN GREEN AND DIGITAL TRANSITION 2021 (HORIZON-CL4-2021-TWIN-TRANSITION-01)
Topic	HORIZON-CL4-2021-TWIN-TRANSITION-01-05 Manufacturing technologies for bio-based materials (Made in Europe Partnership) (RIA)
Consortium	17 Organizations. 15 EU Member States + 2 non-EU state

GREEN-LOOP Consortium Partners

	Partner	Acronym	Country
1	IDENER RESEARCH & DEVELOPMENT	IDE	ES
2	NATIONAL INSTITUTE OF CHEMISTRY	NIC	SI
3	SLOVENIAN NATIONAL BUILDING AND CIVIL E. I.	ZAG	SI
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Executive Summary

This deliverable entitled D5.6 - Report of Wood Composite Bearings (WCB) properties from tribological evaluation for tooling adaptation (injection unit) is the result of task T5.5 as well as related Tasks in WP5. After submission of a first report (D5.5) at a rather early stage of the project, further investigations followed, in which quite promising results were obtained. In the first deliverable (D5.1) of WP5, materials specifications for a possible general use as sliding bearings were summarised. As a starting point, LBRT was regarded as a representative end user for sliding bearings in conveyer rolls and will test wood composite sliding bearings supplied by FHF. Moreover, a further company, Interroll AG, Germany, was involved in some discussions. Therefore, there is a real opportunity for targeted development of wood composites materials for technical applications with moderate tribological loads, which will be an ideal representative use case for wood composite bearings.

To develop the bearing prototypes, suitable composite material compositions have been identified and are in further processing to manufacture prototypical slide bearing for demonstration of performance and feasibility. Three subsequent series (“batches”) of materials were synthesised in WP5. In T5.5 a high number of wood composite material compositions were tested and compared in tribological tests using easy geometries. Moreover, 5 benchmark materials based on polyamide (PA4.6) were tested. The results show that some wood composites have astonishingly low friction coefficients in unlubricated tests. The wear rates were in an acceptable range. Further tests with selected material types showed, that oil lubrication may slightly improve the tribological performance. In further tests with stepwise increased loads and velocities, first results on tribological load capacities were obtained. Based on these results, two promising material types were identified to produce wood composite bearings that will be tested in lab-scale test rigs and in application related tests in WP6.

<u>Finished activities within T5.5:</u>

Set-up tribo-tests with selected standards and selection of polymer benchmark materials.

Tribological testing of WC samples derived from three material batches and benchmark samples.

<u>Ongoing Activities:</u>

Comparative testing at varying loads.

Comparative testing of materials produced using microwave heating.

Preparation of slide bearings for lab tests.

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Abbreviations

AI – Artificial Intelligence

DMP – Data Management Plan

DR – Air flow (%)

DSC – Differential Scanning Calorimetry

FTIR – Fourier Transform Infrared Spectroscopy

GA – Grant Agreement

I_{cl} – Clothes' Thermal Resistance ($m^2 \cdot °C/W$)

ICT – Information and Communications Technology

ISO – International Organization for Standardization

ISO VG - International Standards Organisation Viscosity Grade P_a – Partial pressure of water vapour in air (kPa)

PMV – Mean Expected Vote

PPD – Expected percentage of dissatisfied

RH – Relative Humidity

SEM – Scanning Electron Microscopy

T – Task

T_a – Forced ventilation room temperature (°C)

T_g – Globe temperature thermometer (°C)

T_o – Operating temperature (°C)

T_r – Average radiant temperature (°C)

T_w – Forced-air wet bulb temperature (°C)

TG – Thermo-Gravimetry

TU – Turbulence intensity

v_a – Air speed (m/s)

WC – Wood Composites

WCB – Wood Composite Bearings

WP – Work Package

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1. Introduction

1. Purpose of this document

The purpose of this document is to describe the progress in developing and evaluating wood composite (WC) materials for use in tribological applications, especially for slide bearings. It is summarised, which WC composite materials have been synthesised. The reported results focus on the tribological properties that have been obtained in friction and wear standard tests.

Based on these tribological investigations, a pre-evaluation of different material types was made. The goal was to identify and select promising material types. Moreover, recommendations should be given for further improvement of the tribological properties, but also identify possible risks and drawbacks of the materials. This pre-selection of material types helps focus further research work to enhance efficiency in the production by microwave technologies and reduce the efforts for further investigations, including materials testing and analyses, prototype testing of bearing components and, eventually, assess environmental aspects like recyclability and CO_2 footprint.

1. Need for new materials to mitigate environmental footprints

As already reviewed in D5.1, steel, polymers, and other materials are currently being used by the bearing industry for the manufacture of different bearing components. These bearing materials undergo different heat treatments and processes to attain the desired properties to maximize bearing life and performance. However, this leads to the emission of tonnes of CO_2 for every tonne of steel produced, plus the usage of heavy metals (mainly Pb, Cd, Sb, Cu and Sn) which are used in metallic bearings and are extremely dangerous for the environment. Now, there is no possibility to reduce the carbon footprint of conventional bearing materials.

From a greener perspective, the use of biobased materials aims to reduce the environmental footprint of the manufacturing process of these materials, reuse wood waste from other industrial or natural sources, and reduce maintenance costs. This goal can be reached by using materials that are derived from renewable sources like wood, with the added benefit of reducing the environmental impact related to its manufacture and improving their circularity.

2. General Workflow

The general workflow in this project is visualised in fig. 1 (reproduced from the project proposal). To develop a wood composite value chain, the work starts with the selection of base stocks for bio-based composite materials. The development of promising materials is performed in parallel to the definition and preparation of a microwave-enhanced extrusion process to realise high production efficiency and high material quality. Despite a focus on tribological applications, mechanical testing and several analyses methods are necessary to assess the material quality. Tribological investigations will be performed on three levels: Lab-scale friction-and-wear testing of materials; lab-scale testing of slide bearings of standardised shape; demonstration of slide bearing functionality and performance in a technical application. Finally, a TRL6 will be reached by demonstration of the performance of bearing prototypes.

Moreover, concepts for circularity e.g., by re-use of bearing components milling and re-shaping of WC will be discussed in the project within WP6.

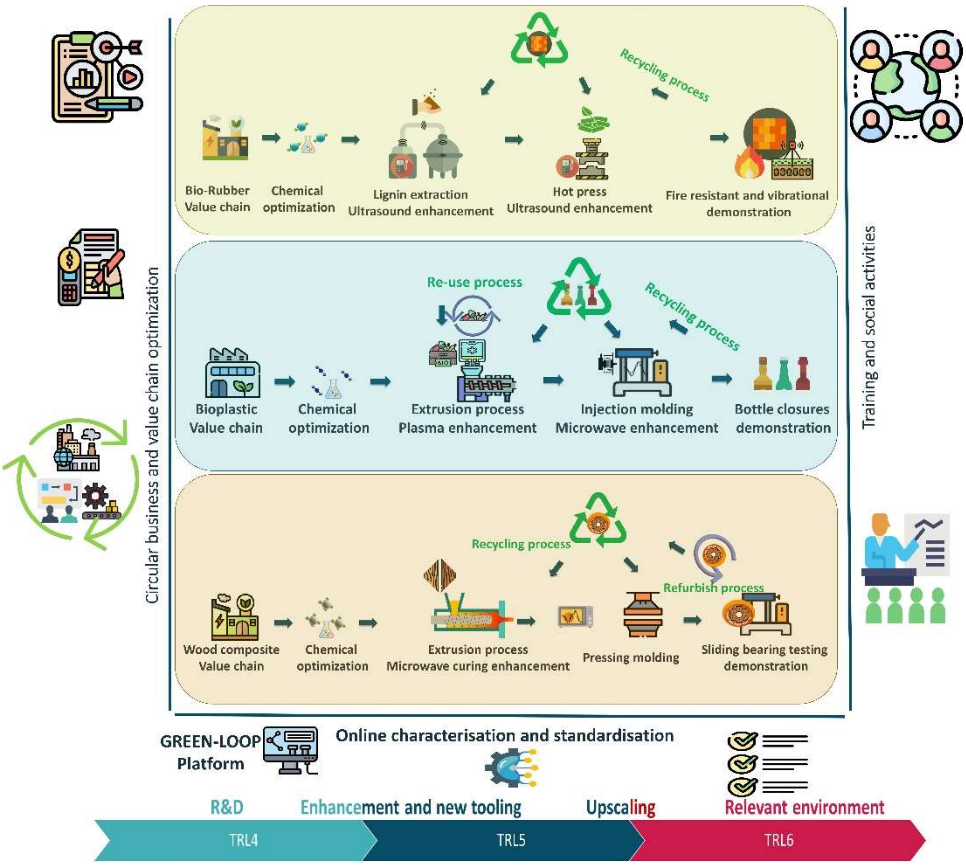

Figure 1: Graphical abstract of the Green-Loop project proposal (reproduced from full proposal)

2.2 Standardization

The biomaterial used for the sliding bearings is be produced in accordance with the following standards (in accordance with Annex A of the deliverable D5.1):

- ASTM D6866
- EN ISO 14125:1998
- EN ISO 1183:2019
- EN 15534-1:2014-04
- ISO 4378
- ISO 6691:2021
- ISO 4178-2

Further explanations for these standards are included in the deliverables D2.4 and D2.5: “Standardisation landscape for biomaterials”.

2.3. Data management

The management of this WP’s data will involve several tasks from this and other WPs: task T1.4. “*Inline monitoring and quality controls*”; task T3.3. “*Upgrades and modifications of equipment in manufacturing lines*” and task T7.6. “*Open science and Data management plan*”.

Throughout the duration of the project, IRIS coordinates the collection of data from the three manufacturing lines onto the ICT (Information and Communication Technology) infrastructure that will be connected to the GREEN-LOOP platform for the purpose of optimizing the entire value chain, as well as the market analyses and identification of replication cases performed in WP7.

Additionally, to comply with the GDPR, RWTH AACHEN is working on a Data Management Plan (DMP) that establishes the guidelines for data management (including the data collected, the data processed in the GREEN-LOOP platform, and the results obtained) throughout their entire lifecycle. This information was published during M06 in deliverable D7.11 "*DMP and open sourcing approach*".

2.4. Circularity

In total, the activities performed in GREEN-LOOP aim to achieve a reduction in CO_2 emissions by 28% and waste material by 40%. To assure circularity and low environmental impact of the products it is necessary to develop solutions either to refurbish bearing components for reuse or to recycle the materials and produce new bearings from recycled base stocks.

A detailed list and a more deepened discussion were presented in D2.1 *“GREEN-LOOP circular economy evaluation*”.

2.5. Modelling

Modelling is carried out by NCC as per task T5.3 “*Modelling, Eco design and manufacture of wood-based composite materials*”, supported by AI models from task T2.5. All materials are characterized by several methods such as TG/DSC, FTIR, SEM and grain size distribution. Samples and components with simple geometries are manufactured by extrusion and press moulding. Quality controls on the WC samples are performed by FHF and NCC by using non-destructive methods such as x-ray or ultrasonic.

2.6. Manufacture

The manufacturing of WC samples has been performed by FHF in collaboration with NCC and UBRIS (T5.4). A multistep process was developed including mixing of raw materials, extrusion and final press moulding of even plates. All extrusion trials were accomplished with electrical heating system and many compounds using different raw materials were investigated. The extrusion process was performed and optimized by FHF using bio-based raw materials to get pellets. These pellets were further pressed into plates. Samples for tribological testing and mechanical characterization were derived by accurate machining of such press moulded plates. Mechanical testing and microstructural characterisation of WC were carried out in T5.4 and are reported in D5.4.

The compression moulding is carried out by FHF to produce first lab samples. For the further upscale, NCC is using a hydraulic press and metallic tooling to produce panels. The panels will be machined into the correct geometry for the slide bearing as defined above. The panels are pressed at the NCC using a HARE press. More manufacturing details can be derived from D5.3.

2.7. Microwave Enhancement

To increase energy efficiency during the production of the WC base stock, a MW device was developed and implemented in the process chain at FHF. The progress of this development is reported in D5.2. The WC materials that have been tribologically examined so far, were not yet produced with microwave enhancement. In M23 the MW heating could be successfully implemented into the extruder system and first extrusion trials at FHF showed the viability of the new assembly. Moreover, the power meter derived from IRIS was installed and connected to measure the energy consumption. This data will be transferred to the ICT platform to further calculate the reduction of energy consumption when using the MW heating system.

2.8. Tribological testing and evaluation

Lab scale tribological testing of WC bearing materials and components are performed by FHF (T5.5), this will include basic materials tests like Pin-On-Disc-Tests to determine friction coefficients and wear characteristics. These tests enable screening of materials to identify most promising candidates for bearing materials, investigate tribological mechanisms and give advice for further materials optimisation possibilities.

For component tests, a basic bearing geometry based on standard cylinder shape will be used. These tests will be performed to obtain information about general tribological behaviour und application-like conditions and thus evaluate the possibilities and limitations of WC for slide bearings, in general.

2.9. Demonstration

The products will be tested in an industrially relevant setting at LBRT’s facilities. The sliding bearing will be substituted in one of the available injection moulding machines to test their performance during the production of bottle closure. This demonstration overlaps with WP6 as one or more production cycles will produce samples with the processes.

Iterations with T5.4 and T5.3 will be done to ensure the proper results at TRL5.

3. Results and Discussion

3.1. Requirements

General specifications for bio-based WC for tribological use were defined in D5.1 according to the current state and characteristics of state-of-the-art polymer materials in use are given in the following table.

Table 1: Technical requirements for the wood composite final product

Product	**Wood Composites**	
Physical Properties	**Value**	**Units**
Density	Not relevant	-
Micro-porosity	Favorable to retain lubricant in the sliding contact	-
Water absorption	Acceptably low	-
Mechanical Properties	**Value**	**Units**
Tensile strength	> 50	MPa
Compressive strength	> 70	MPa
E-Modulus	> 1000	MPa
Thermal stability	Up to 150	°C
Tribological Properties	**Value**	**Units**
Wear Coefficient	$< 10^{-5}$	$mm^3/(Nm)$
Friction coefficient	< 0.1 under lubrication	-
	< 0.3 under dry sliding	
pv-value	> 0.3	MPa · m/s
Working conditions	**Value**	**Units**
Temperature	0 - 100	°C
Lubrication	Oil, grease	-
Standards to address		
ASTM D6866, EN ISO 14125:1998, EN ISO 1183:2019, EN 15534-1:2014-04, ISO 4378, ISO 6691:2021, ISO 4178-2		

From a current point of view, the previously defined requirements for tensile strength, friction coefficient under lubrication and the oil or grease lubrication appear to be less important, since tensile strength is usually not needed and not specified for composite materials for use as slide bearings. The reason is that there are no critical tensile stresses in such applications. This means that materials with low tensile strength will be generally acceptable, if the compressive strength is in a range of 70 MPa+. Moreover, the main advantage and benefit of such bearing composite materials is their ability to work without lubrication. Therefore, for the requirements that are marked in orange, deviations from the defined specifications can be accepted.

3.2 Materials

The following table shows all WC material batches that have been produced at FHF (ISC) and delivered for further characterisation. So far 3 material batches have been produced containing several biopolymers as a matrix and varying amounts of wood fibres, glass fibres and other additives as fillers. In total 37 experiments (see Table 2) were performed.

Samples derived from batch 1 (V205-V219) are based on commercially available biopolymer wood fibre blend (type A) which was doped with graphite as the filler. The experimental materials V222-V230 also derived from batch 1 were investigated in milestone 6 by IDE to investigate their physical interaction with the microwave heating.

In batch 2 (V231-V244) a novel biopolymer (type B) in combination with several fillers based on lignin, glass powder and glass fibres were investigated to increase the mechanical and thermal stability of the wood composite.

In batch 3 (V259-281) biopolymers from type B and type C as well as glass fibres as fillers in different concentrations were investigated to further improve the strength and tribological properties.

Most of these WC material types were tested tribologically at FHF (IWM) within T5.5 and mechanically tested at ISQ within T5.4. The physical-chemical properties of most raw materials were determined by ISQ and UBRIS including all used matrix types (A-C), lignin, wood fibres/particles as well as glass powders/fibres.

Table 2: Produced wood composite materials derived from 3 series of materials synthesis (batch 1-3) and their codes

Material Code	**Batch Nr.**	**Destination**	Material Code	**Batch Nr.**	**Destination**	Material Code	**Batch Nr.**	**Destination**
V205	1	IWM	**V231**	2	IWM	**V259**	3	IWM/ISQ
V206	1	IWM	**V234**	2	IWM	**V261**	3	IWM/ISQ
V207	1	IWM	**V237**	2	IWM	**V263**	3	IWM/ISQ
V208	1	IWM	**V238**	2	IWM	**V265**	3	IWM/ISQ
V211	1	IWM	**V240**	2	IWM	**V267**	3	IWM/ISQ
V212	1	IWM	**V243**	2	IWM	**V269**	3	IWM/ISQ
V213	1	IWM	**V244**	2	IWM	**V271**	3	IWM/ISQ
V214	1	IWM				**V273**	3	IWM/ISQ
V215	1	IWM				**V275**	3	IWM/ISQ
V216	1	IWM				**V277**	3	IWM/ISQ
V217	1	IWM				**V279**	3	IWM/ISQ
V218	1	IWM				**V281**	3	IWM/ISQ
V219	1	IWM						
V222	1	ISQ/IDENER						
V227	1	ISQ/IDENER						
V228	1	ISQ/IDENER						
V229	1	ISQ/IDENER						
V230	1	ISQ/IDENER						

As benchmark materials, various types of PA4.6-based polymer composites were used that are listed in table 3. PA-based materials are very often used for polymer slide bearings. Therefore PA4.6 was identified as a common polymeric state-of-the art material to compare it with the new developed wood composites. All benchmark materials were supplied by the company DSM.

Table 3: Benchmark materials, supplied by company DSM

Material	**IWM name**	**Composition**
StanylTW341	TW341	PA46
StanylTW371	TW371	PA46 + PTFE
StanylTW200F6	TW200F6	PA46 + 30% GF
StanylTW271F6	TW271F6	PA46 + PTFE + 30% GF

3.3. General Shape and sample preparation for mechanical and tribological tests

The manufacture of the wood composite materials is described in section 2.6 and 3.2. Samples for tribological tests were prepared from extruded plates derived from various material types (see table 2). Benchmark materials (see table 3) were supplied by the company DSM in the form of plates to prepare samples with the same shape as the wood composite materials. The sample geometry is shown in Figure 2.

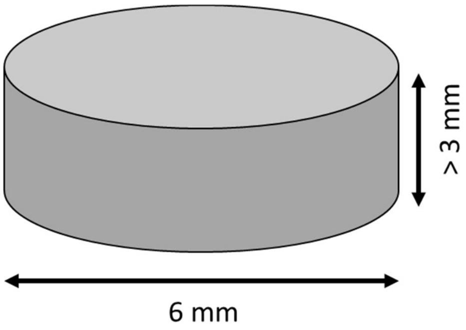

Figure 2: Sample geometry for tribological lab test

3.5. Tribological tests and test conditions

Tribological tests were conducted using a pin-on-disk tribometer (TRM100, Wazau, Germany). New sample holders were designed to fix the sample shapes (s. Figure 2) against standard bearing rings made of 100Cr6 steel (Hardness HRC > 60). Pictures of the tribometer and the test setup are shown in Figure 3. In this tribometer, the rotating shaft with the steel rings as counter materials is on the upper side. The composite samples are fixed on the lower side.

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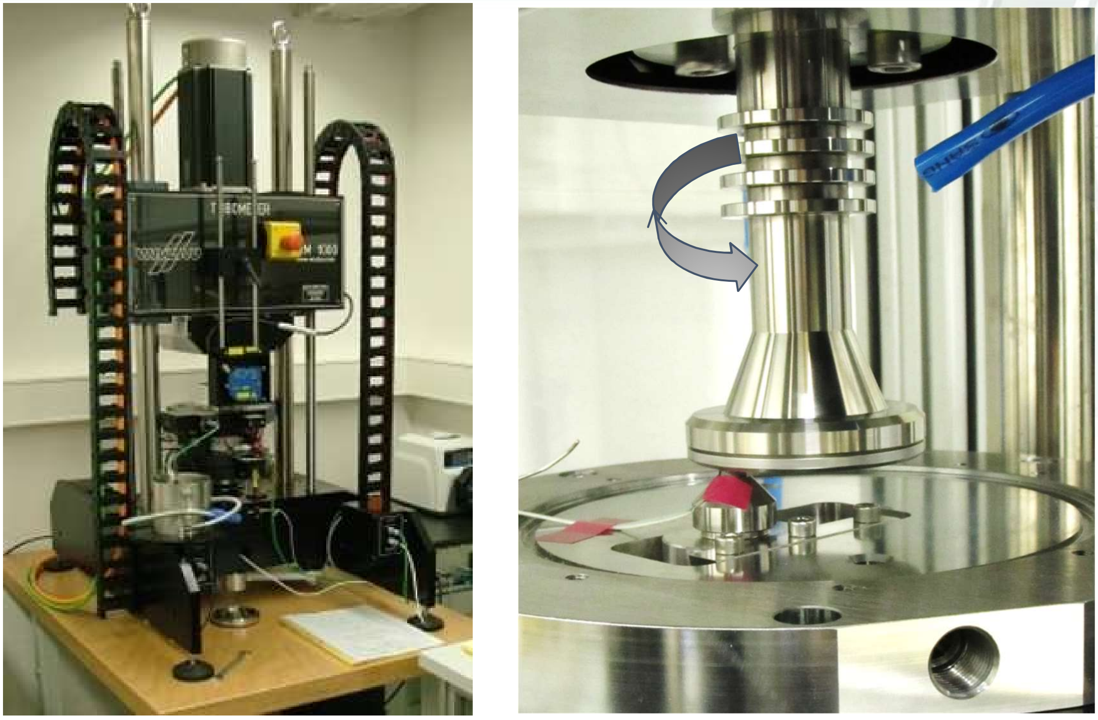

Figure 3: Tribological test rig (TRM 1000, Wazau, Germany) and test setup.

The test conditions of this test series are given in table 3.

Table 4: Test conditions for comparative tribological tests

Normal force	85 N
Contact pressure	3 N/mm²
Sliding velocity	0.1 m/s
Temperature	room temperature
Test duration	6 hours
Lubrication	dry (air at medium rel. humidity)

4. Results and discussion

4.1. Material properties

Materials for sliding bearing must have an acceptable compressive strength, high thermal stability and density. At FHF compression strength tests were performed by using standard ISO604. Test samples should fulfil the required compression strength level of > 70 MPa and Youngs Modulus > 1000 MPa.

Samples of batch 1 (V205-V219) revealed strength values up to 95 MPa and Youngs Moduli < 1000 MPa and could fulfil the requirements. Specimens from the second and third batch also showed sufficient compressive strength. Therefore, mechanical properties are rated as sufficient for general use in slide bearings for moderate tribological loads.

Further results on material properties are within the scope of Task 5.4 of the project and will be reported in deliverable D5.4.

4.2. Evaluation of friction and wear results

4.2.1 Comparative friction and wear tests

Because of the large quantity of results, only a selection of representative results can be given at this place to depict the progress of the development and its appraisal.

For materials from the first batch (V205 to V230), friction and wear results were not sufficient. These materials contained a resin as matrix with insufficient thermal stability. Due to high friction in the model test, frictional heat caused temperature increase beyond the thermal stability and therefore excessive wear. Therefore, biopolymers with higher thermal stability were chosen as a base material for the second material batch (V231 to V244). In addition, glass fibres were used as a further filler to increase the wear resistance of the composites. Lignin and graphite that had been used as fillers in batch 1 and batch 2, were identified as not promising.

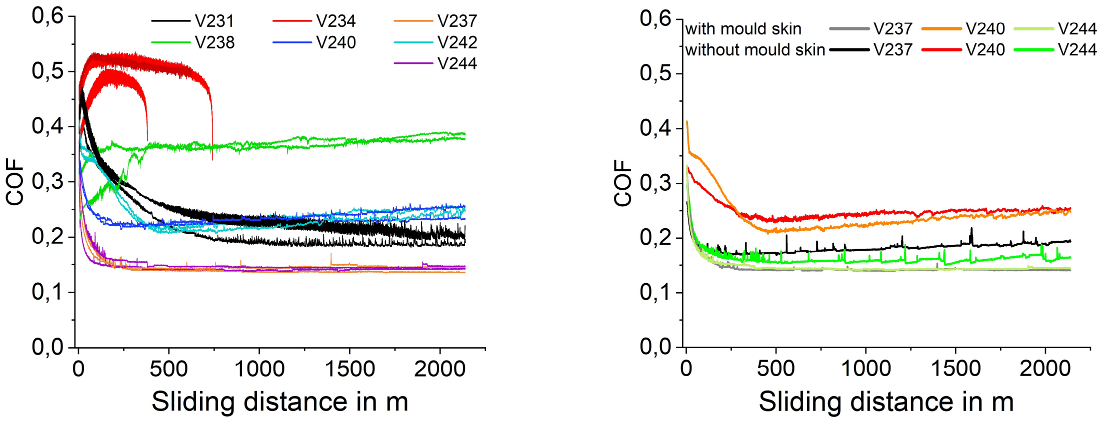

Figure 4: Friction coefficients (COF) over sliding distance of loop 2 materials; right hand side: Selected results with and without mould skin.

Figure 4 shows results of tribological pin-on-disc tests for material types from batch 2. For each material, at least two identical tests were conducted. The results are well reproducible and show that except V234 and V238, all composites reached rather low friction values. The resulting friction values were quite different and ranged from 0.15 to 0.5. Lowest friction coefficients were obtained for V237 and V244. Both materials are composed of a bio-based polymer with only wood fibres and glass fibres as fillers. To check, if the mould skin of the samples has an influence on the friction behaviour, several materials were tested with different surface finish. The results in Figure 4 (right diagram) show that no significant influence is observable.

After these promising results for the materials in batch 2, further variations of the materials compositions were made in batch 3. In most cases only the filler amounts were varied, and in some materials (V265, V267, V269, V277) glass powder was used instead of glass fibres. V273 contained 10 % of lignin. For V275 and V277, biopolymer C was used as a basis. As can be seen in Figure 5, almost all materials of batch 3 showed very low friction coefficients. Exceptions are those materials (V275, V277) for which biopolymer C was used. Slightly higher friction was also observed for V279 that did not contain any glass fibres.

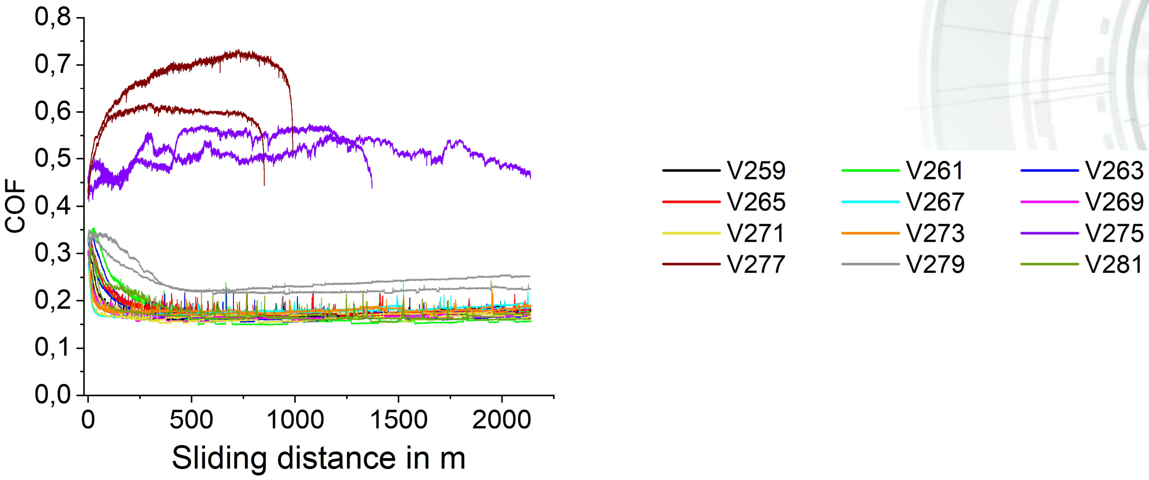

Figure 5: Friction coefficients (COF) over sliding distance of loop 3 materials.

Compared to these very low friction values, the benchmark materials all resulted in significantly higher friction values in the range of 0.26 for PFTE-filled PA4.6 and 0.55 for unfilled or glass fibre-filled PA4.6.

In Figure 6, the results of the tribological test of all batch 2 materials are summarized in a two-dimensional field, in which the mean friction coefficients (x-axes) are combined with the wear results (y-axes). This diagram impressively shows that very low friction values are obtained with some specific compositions. Moreover, the wear coefficients are lower than 10^{-5}, which was specified as KPI in D5.1. In direct comparison with the benchmark materials, friction coefficients are significantly lower, and wear coefficients are in the same range.

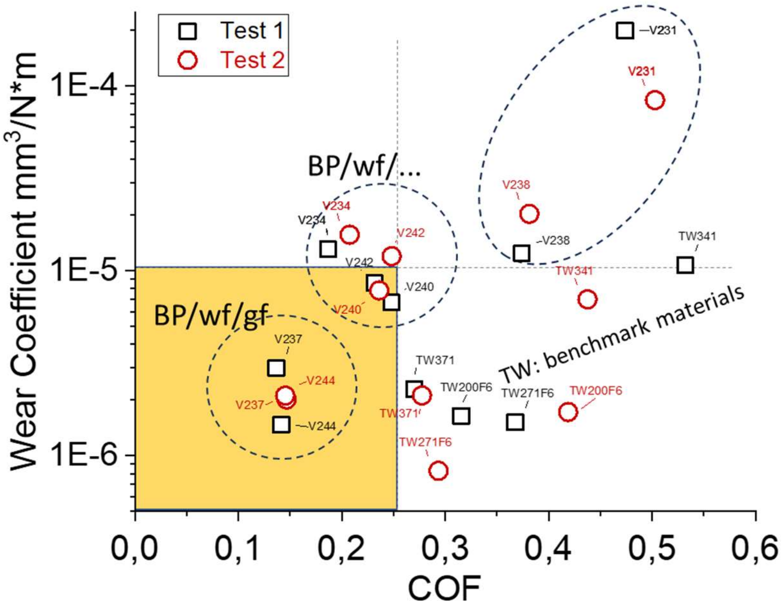

Figure 6: Overview of friction and wear results of batch 2 materials. Wear coefficient is defined as Volume Loss in mm³ divided by Normal Force in N and sliding distance in m.

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Based on these results for the materials of batch 2, further materials with comparable compositions were produced, in which the wood and glass fibre content was varied. There results for these batch 3 materials are summarised in Figure 7. Except two material types, in which a different bio-based polymer was used as a matrix, all materials resulted in friction and wear values that are located well within the region of acceptably low friction and wear.

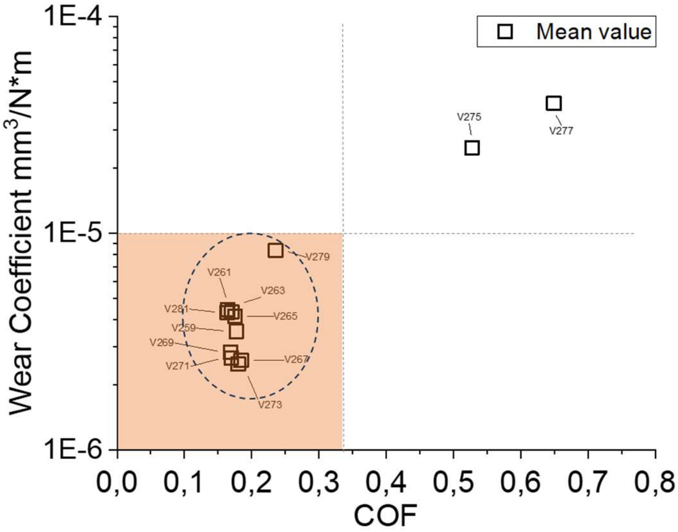

Figure 7: Overview of friction and wear results of batch 3 materials. Wear coefficient is defined as Volume Loss in mm³ divided by Normal Force in N and sliding distance in m.

4.2.2 Lubricated tests

To investigate the influence of oil lubrication on the tribological behaviour of WC, three bio-based oils without any tribological additives were selected. Tests were conducted using the same test parameters as in the unlubricated tests.

Some test results for three different WC materials are shown in Figure 8igure 8. These results indicate that no significant improvement of the frictional behaviour could be reached in the first trials.

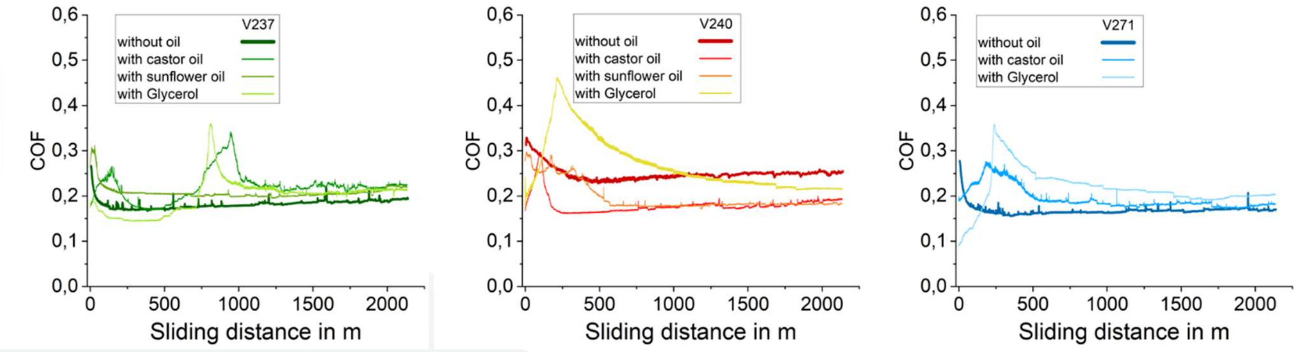

Figure 8: Friction coefficients of selected tests using natural oils as lubricants.

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To reach a better effect of oil lubrication, further materials modifications would be necessary to improve oil retention in the material microstructure und wettability. Moreover, real technical lubricants that contain additives like friction modifiers, extreme-pressure additives, dispersants etc. would be necessary. This would, however, worsen the possibility to recycle the materials to establish a circular economy. On the other side, a real technical strength and benefit is the excellent dry running behaviour that has been observed so far (see section 4.2.1). It was therefore decided to follow that track for unlubricated technical applications of slide bearings.

4.2.3 Load capacity

The load capacity of a selection of WC was examined in further tests, in which the load and the sliding velocities were stepwise increased. Representative results of these tests are shown in Figure 9. It is noticeable that these materials had different friction coefficients at the beginning of the tests at low loads and sliding velocities. At low velocities and enhanced load, the material V240 showed decreasing friction values to values lower than 0.2. However, at higher sliding velocities, both materials showed increasing friction coefficients. At higher velocity and higher load, the test was stopped due to excess wear. This indicates that the use of the WC should be restricted to medium loads and velocities. These results were expected and rated as acceptable, since the load capacity of such composite materials is mainly governed by the thermal stability of the composite matrix. To reach higher valued (which is not the target now) a biopolymer with higher thermal stability would be needed.

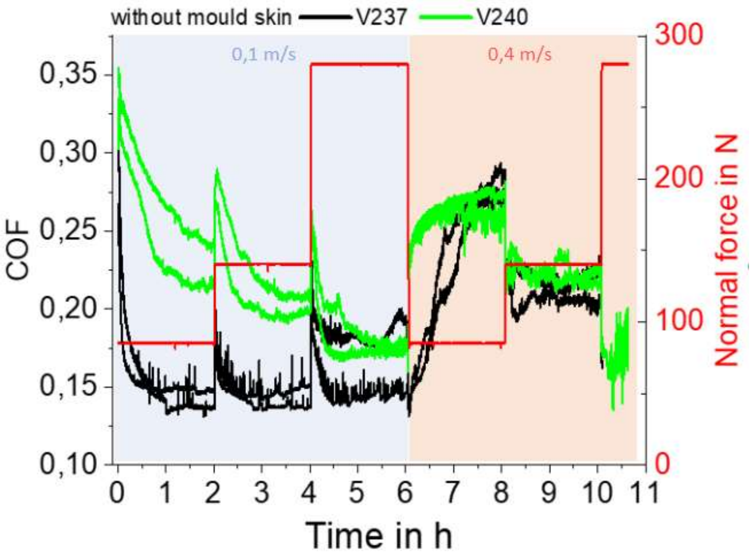

Figure 9: Friction coefficients in stepwise increased load-velocity ramps. The background colour indicates the sliding velocity (0.1 m/s and 0.4 m/s) the red line shows the normal forces which were stepwise increased.

5. Future workflow and Upscaling

The possibilities for the implementation and use of WC slide bearings were discussed for the demonstration of the project suitability of WC for slide bearings. Promising material types were identified and selected for further evaluation. In addition, material samples produced using microwave heating will be tested within WP6 and evaluated to ensure a similar material quality. Therefore, the ongoing activities in T5.5 will be comparative model tests at varying loads, comparative testing of materials produced using microwave heating, and preparation for component tests for slide bearings for lab tests.

The further development of the WC bearings will follow the work description of the of the Grant Agreement (GA) as well as the general workflow described in section 2 of this report. This will include materials and manufacturing development, lab-scale analysis, and testing activities. After M25 the activities will be transferred to WP6 to develop concepts for process scale-up, circular economies and demonstration of the suitability of WC bearings in conveyers.

6. Conclusions

After defining the specifications for WC materials further work was performed according to the work plan of Green-Loop. The specifications were used as a guideline for materials development and tribological assessment (see table 1).

Recent results on tribological behaviour have been very promising and may be regarded a success. These promising characteristics include suitable mechanical material properties, very low friction coefficients without the use of liquid lubricants, and acceptably low wear rates. With respect to the general material requirements, all important materials specifications have been fulfilled.

Finally, the last task within WP5 will be to finish the evaluation of the tribological performance of the WC materials and bearings on a model scale and to prepare for component testing with real bearing geometries to demonstrate their feasibility for technical use.

Funded by
the European Union