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Perennial biomass crops as sustainable feedstock for carbon dioxide fixation in building materials

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Referent: Prof. Dr. Ralf Pude
Koreferrentin: Prof. Dr. Margit Schulze

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Preamble

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1.3 List of Abbreviations

Abbreviation	Meaning
ANOVA	analysis of Variance
BLC	bonded levelling compound
CO_2	carbon dioxide
C_3 plant	plant that utilizes the C_3 photosynthetic pathway
C_4 plant	plant that utilizes the C_4 photosynthetic pathway
CP	Cup Plant
CV	coefficient of variation
CNC	cellulose nanocrystals
DIN	German industry standard
EU	European Union
EN	European Norm
EPS	expanded polystyrene
EtOH	ethanol
FA	formaldehyde
Fmax	maximum force
FeMax	length according to Feret

FeMin	width according to Feret
FC	Folin-Ciocalteu
FTIR	fourier-transform-infrared
GPC	gel-permeation chromatography
ha	hectare
HSD	honest significant difference
ISO	international organisation for standardization
LCA	life cycle analysis
l/w	length to width ratio
MOE	modulus of elasticity
MOR	modulus of Rupture
MDI	methylendiphenyl-diisocyanat
MA	maleic acid
n	sample size
NaOH	sodium hydroxide
PLA	polylactic acid
PHBV	poly (hydroxybutyrat-co-hydroxyvalerat)
PBAT	polybutylene adipate terephtalate
PBS	polybutylene succinate
PVA	polyvinyl alcohol
PVAc	polyvinyl acetate
p	P-value
PP	polypropylene
PLLA	poly lactic acid
PE	polyethylen
rpm	revolutions per minute
spht	sphericity index
TS	thickness swelling
WA	water absorption
wt%	weight percent
Xarea	equivalent circle radius

2. Summary of the Publications

2.1 Zusammenfassung

Das Ziel einer biobasierten Kreislaufwirtschaft besteht darin, die Abhängigkeit von fossilen Ressourcen und Treibhausgasemissionen zu verringern. Mehrjährige Biomassekulturen (PBC) können Vorteile bieten, wie z. B. einen geringeren Bedarf an Dünger, der den CO_2-Fußabdruck durch ein hohes Verhältnis von Ertrag zu Inputs verringern kann. PBC werden derzeit hauptsächlich in der Bioenergieproduktion eingesetzt, da es an entwickelten Anwendungen für höherwertige Produkte mangelt. Die Materialproduktion für die Bauindustrie trägt zu 9 % der globalen CO_2-Emissionen bei und lohnt als Ziel für die Umstellung auf mehr biobasierte Materialien. Eine Literaturstudie an *Miscanthus* beschreibt beispielhaft mögliche höherwertige Anwendungen in Polymerverbindungen und Baumaterialien. Durch Unsicherheiten in der Eignung der PBC als Ausgangsmaterial wurden die Literaturergebnisse als Grundlage für die experimentelle Herstellung und Bewertung von selbstbindenden Faserplatten und biobasierten Leichtzuschlägen für Leichtbeton übertragen.

Bei selbstbindenden Faserplatten zeigte die Partikelgrößenverteilung und die Art der Biomasse (*Picea*, *Paulownia*, *Miscanthus*) erheblichen Einfluss auf Materialfestigkeiten. Drei Biomassen wurden mit einer Hammermühle zu selbstbindenden Faserplatten verarbeitet, wobei der Feinanteil angepasst wurde. Biomassespezifische Unterschiede bei zunehmendem Feinanteil und individuelle lineare Beziehungen zwischen dem Elastizitätsmodul und der erreichten Plattendichte wurden beobachtet. Kombinationen aus verwendeter Biomasse und Feinpartikelgehalt erfüllen die Elastizitätsmodul Anforderungen für tragende Anwendungen, während das Bruchmodul verbessert werden muss. Die Dickenquellung in Wasser ist unzureichend hoch, aber Konzepte für eine biobasierte und potenziell recycelbare hydrophobe Behandlung werden in laufenden Arbeiten werden entwickelt.

Polystyrol angereicherter Leichtbeton mit reduzierten Festigkeitsanforderungen und definierten Dämmeigenschaften wurde ausgewählt, um Parenchymreiche *Silphie* als teilweisen Ersatz für die dämmenden Leichtzuschläge aus Polymerschaum zu testen. Zwischen mineralischen Bindemitteln mit Zement und hydraulischem Kalk mit erhöhter Biomasseverträglichkeit wurde festgestellt, dass ein erhöhter w/c-Wert einen hohen Grad an Biomasse-Substitution von 45 wt% erfordert, um akzeptable Druckfestigkeit und gute λ-Dämmwert zu erhalten.

Prozess- und Stoffstrom Optimierungen und die Erfassung von Lebenszyklusanalysen der Produkte müssen erfolgen, um das Einsparpotenzial von CO_2 zu ermitteln. Die Bestimmung der für eine Anwendung relevanten Rohstoffqualitäten und entsprechende Prozessoptimierungen sollten auch mit anderen Biomassen erfasst werden. Dies kann potentielle Rohstoffquellen für eine biobasierte und kreislauforientierte Wirtschaft ermitteln.

2.2 Summary

The aim of a bio-based and circular economy is to help reduce dependence on fossil resources and greenhouse gas emissions. Perennial biomass crops (PBC) can offer advantages such as low fertilizer requirements which can reduce the embedded carbon footprint by a high output to input ratio of the biomass. However, PBCs are currently mostly used in bio-energy production due to lack of developed higher valued product applications. Material production for the construction industry contributes to 9% of global CO_2 emissions and is therefore a worthwhile target for switching to more bio-based materials. A literature study on the example biomass *Miscanthus* in materials applications describes possible routes for higher valued application in polymer compounds and construction materials. Due to uncertainties in the feedstock suitability of the PBC the literature results were transferred as basis for experimental material production and performance evaluation of self-binding fiberboards and biobased light aggregates for vegetal light concrete.

For self-binding fiberboards, particle size distribution and biomass type (*Picea*, *Paulownia*, *Miscanthus*) significantly influenced material strength in the dry hot pressing process. Three biomasses were processed into self-binding fiberboards using a hammermill, with adjustments to the proportion of fine dust. Biomass specific sensitivities to an increasing amount of fines and linear relationships between the modulus of elasticity and the board density achieved are observed. Specific combinations of used biomass and fine particle content satisfy the modulus of elasticity requirements for load bearing applications while the modulus of rupture requires improvement. The thickness swelling in water is insufficiently high in the reported results, but in ongoing research projects concepts for a bio-based and potentially recyclable hydrophobic treatment is beeing developed.

Based on the literature study polystyrene-enriched lightweight concrete with reduced strength requirements and defined insulation properties was chosen to test parenchyma-rich *Silphie* as a partial substitution for the polymer foam lightweight aggregates. Using two mineral binder formulations for cement and hydraulic lime with increased biomass compatibility it is observed that an increased w/c ratio necessitates a high degree of biomass substitution of 45 %wt, to obtain a material with acceptable compressive strength and good λ-insulation value.

Further development can be carried out by optimizing the process and material flow and recording the life cycle analyses of the process and product in order to determine the CO_2 savings potential. The targeted determination of the feedstock qualities relevant for an application and the corresponding process optimization should be recorded together with other biomasses. This can expand the base of raw material sources for a bio-based and circular economy.

3. Introduction

3.1 Potential Perennial Biomass Crops

The reduction of total emitted greenhouse gases is a current global challenge where carbon dioxide (CO_2) is specifically targeted for a reduction to net zero in 2050 [3]. The greenhouse gas emissions of some processes is unavoidable and CO_2 sequestration technologies are being suggested to obtain CO_2 sinks. The growth phase of plants presents a temporary CO_2 sink by uptake of atmospheric CO_2 and light in photosynthetic pathways to build up the plant's biomass. Biomass contains approximately 48% Carbon [4,5], thereby storing approximately 1.7 kg of CO_2 per kg of used biomass until either biological or thermal decomposition occurs.

Bio-economy is the application of biomass for economic purposes which often includes cultivation of plants for specific applications. Applications of plant-based biomass are mostly food, fodder, and bioenergy and take up 95% of 11,7 million ha of cultivation area for agricultural crops in Germany [6]. Harvested biomass contains nutrients which are especially relevant to food and fodder. Some of the nutrients carried off need to be resupplied to the soil to ensure plant growth in the next iteration. Different elements need to be available to substantiate stable yields and are commonly supplied as fertilizers on agricultural land. The production and application of fertilizers causes emissions [7–9]. The amount of agronomical input in form of performed work in cultivation and especially fertilizers differs between biomasses, thereby causing disparities in the associated emissions and stored CO_2 between different biomass cultures [7, 10].

Perennial biomasses can offer sustainability benefits especially in the CO_2 footprint [7–9, 11, 12]. Perennial plots can be cultivated for decades, so the replanting cycle is longer and the associated costs and agronomical input can be reduced compared to annual plants. By undergoing senescence, perennials can translocate nutrients to the root system or rhizomes, reducing the need for fertilizers improving the input/output ratio [8, 13]. Due to the established root system the plant growth starts earlier in the vegetation period, leading to taller shoots suppressing weeds such that herbicide application becomes unnecesary in established dense growths. Sufficiently deep root systems can reach deeper water or moisture layers and may be less susceptible to seasonal drought, especially in the early growth phase [14].

A non food application of biomass is the farming for energy plants to substitute fossil fuels with currently 2.2 mio ha in germany [6]. However, the application of biomass to produce energy and reduce the emission of new CO_2 by substituting fossil fuels is currently being critizised for the land use change. The conversion of biomass to energy is also a short life cycle without substantial carbon storage capacity.

Industrial crop applications are actively pursued to a smaller share of 279000 ha of cultivation area in Germany [6]. Increased lifespans in higher valued applications display a longer temporary carbon storage during the product use phase while still displaying their energetic value in thermal disposal. Examples of bio-based industry applications are growth substrates from different crops and residues [15], or biorefineries and the formulation of platform chemicals [16–19], as well as in pulp and fiber production processes [20], and in different construction materials.

The application of bio-based materials in construction has the perceived benefits of a higher economical biomass value and longer CO_2 storage compared to the application biofuels [21,22]. For application in building materials, the nutritional value of biomass becomes irrelevant and other parameters such as the yield and the costs associated with production become base of decision making processes for specific cultivars.

Due to the reduced fertilizer requirements for biomass production perennial biomass crops may present a more sustainable feedstock in terms of embedded energy to be processed to construction materials. Due to quick biomass accumulation and existing farming schemes the three biomasses *Miscanthus*, *Silphium*, and *Paulownia* were deemed to possibly supply a feedstock for development of construction materials.

3.1.1 *Miscanthus*

Miscanthus is a perennial C4 sweet grass (*Poaceae*) [23] with an observed height of up to 4 m [24,25], originating from Southeast Asia [23]. In spring winter dormancy ends and new shoots rise from the rhizomes which stored nutrients [26, 27]. *Miscanthus* generally has a rapid growth and biomass accumulation when sufficient water is available during the growing season [28].

The period for harvesting can be between the end of the growing season in fall and the beginning of the next growing season in spring [26]. Due to the winter dormancy and nutrients relocation, a low input of fertilizer can result in high harvestable biomass output. Output to Input ratios may be as high as 13.6 depending on the precise farming management [8,9,29]. By comparing the environmental impact of different biomasses in a hot spot analysis the largest impact was found to be in biomass cultivation where fertilizer production and application cause the main impact [7]. In the comparison of different bioenergy cropping systems only *Miscanthus* achieved CO_2 neutrality [8]. It is technically feasible to extract two green harvests at the expense of high nutrient exports and thus increased fertilizer costs [30]. A single annual harvest of *Miscanthus* is commonly performed.

The sterile *Miscanthus* × *giganteus* as a hybrid *Miscanthus sinensis* of *Miscanthus sacchariflorus* [31] is the most common commercial cultivar [32]. Due to sterility of the commercial cultivar, rhizome cutting is the common propagation method, involving splitting of juvenile rhizomes from an established field [33]. Seedable varieties and the associated establishment methods are being researched [34] in order to lower the costs of establishment.

This *Miscanthus* $\times$ *giganteus* variety was selected for its high yield of 25 – 30 t ha^{-1}, with the higher yields reported for warmer southern European climates under irrigated conditions [26]. For colder climates, a *Miscanthus sinensis* is preferred for increased cold tolerance [26].

Different varieties may differ in terms of winter hardiness or biomass yields for certain, especially colder, climates [27, 35, 36]. After selecting a site-specific high-yielding genotype maximum yield disparities of a factor of two were found by comparison of two fields in Stuttgart (20 t ha^{-1}) and Moscow (9 t ha^{-1}) [35].

Depending on the intended use, there are different genotypes, selections and hybrids [26].

For some applications, such as bioenergy and methane, an early harvest is desirable due to the high fresh mass with increased nutrient and sugar content which then requires a renewed supply of nutrients as fertilizer to avoid decreased harvest and soil depletion [37]. Due to the perennial character, the fertilizer input can be reduced if a natural nutrient shift takes place during senescence [26, 37]. In addition, ecosystem services such as reduced evapotranspiration rates, and soil quality as well as habitat functions may be found in a late-harvested cultivar [11, 38]. The quality characteristics of the biomass change during senescence [26], so the time of harvest should be chosen deliberately [27] especially so if the ecological impact and CO_2 storage capacity are part of the intended application.

In other applications where sugar and nutrient content are not critical, a later harvest date may reduce the harvested fresh mass due to partial loss of leaves, translocation of nutrients and reduction of moisture in the harvested biomass [27, 35].

Miscanthus has also been proposed as raw material for different applications. It was suggested as lignocellulosic feedstock for industrial applications mainly as source of hemicellulose for fermentation products [30] or as source of lignin or its derivatives as base for platform chemicals [39, 40]. Research into the lignin extracts from different genotypes revealed structural differences between the molecules [39].

It was also proposed as fiber and particle source for different applications such as pulp for paper [41], fiber for insulation [7], substrate replacement [15] [42], in compounding with polymers where the fiber to matrix bond posed the bottleneck to structural reinforcement as phase separation occurred [43–55]. It has also been introduced into different forms of concrete and light concrete, where water absorption to- and release of extractives from the biomass were observed as the main problems to cause a lack of strength increase by fiber addition [2, 56–58]. Attempted strategies involved the addition of water, the silanization of *Miscanthus* and coating of the biomass by slurry [58–60].

Miscanthus has also been applied in particle shape in the production of fiberboards boards. Different strategies with different binders and binder concentrations [61–64] were tested, as well as mixtures with other biomasses [1, 65–67]. Self-binding fiberboards have been accomplisched after variations of pretreatment methodsssuch as steam explosion treatment [68–71]

3.1.2 *Paulownia*

The *Paulownia* is a deciduous tree from the family *paulowniaceae* native to East Asia. The species demonstrates a rapid growth rate in young trees, with an increase in height of 2–4 m observed within a single year [72, 73]. Paulownia trees have the potential to reach a diameter of 2 m under optimal conditions and, in extreme cases, a height of up to 40 – 50 m when mature. However, regular heights of 10 – 20 m have also been documented. It is not uncommon for roots to extend to a depth of 2 m, though in some cases, they may reach even greater depths under optimal conditions [74].

It was introduced to the temperate regions of the eastern United States as a crop in 12- to 15-year rotations in the 1970s [73]. Over the past two decades, the genus has been adapted in southern Europe and the Middle East [74]. As a pioneer species, Paulownia is capable of thriving on marginal soils [73, 75]. The tree is particularly well-suited to Mediterranean climates with high water availability [76].

Due to their rapid growth, biomass accumulation and ability to populate liberated habitats such as train tracks or industrial wasteland, some *Paulownia* species are classified as invasive. This includes *Paulownia tomentosa* in the United States, Austria [74] and Germany [77]. However, it should be noted that the specific regulations that apply may vary depending on the species and the geographical context in which it is situated.

Paulownia may be planted in short rotation coppice [22, 75, 78]. Local legislation may impose restrictions on trees planted in short rotation coppice as is the case in Germany where the lifetime of a short rotation coppice may not exceed 20 year [79]. If the objective is to utilize the *Paulownia* for the production of roundwood, a cycle of 6–15 years with a spacing of 4×4 m^2, is recommended. Conversely, a biomass production cycle can be shorter with an increased density of 2×1.5 m^2 [74, 76]. Dry biomass production can range from 1.5 – 14 t ha^{-1} from the second year onwards, depending on the selected genotype and environmental conditions [74].

Due to potential issues with invasivity an increasing number of plantations are based on hybrids [74]. Seed propagation is common but requires fertile seeds. Plantlets of sterile hybrids can be produced from root cuttings and *in Vitro* propagation by tissue culture. *In Vitro* culture can deliver continuous supply of planting material independent of seasonal limitations as scalable method for industrial production. Research is being done to improve *in Vitro* propagation methods by identifying cells involved in adventitious bud development [80] or in protocol improvements for hybrids such as *P. elongata* × *P. fortunei* [81, 82].

Various hybrids are being selected crossing species with high productivity and stability under different environmental conditions [74] as well increased cold tolerance or survival [83].

Applications where the high biomass productivity is relevant are bioenergy where Paulownia is analyzed for its gross heating value [84], its briquettes and pellet qualities [85] *Paulownia* is also cultivated or researched as potential raw material to substitute for scarce timber, e.g. in Iran [86], Turkey [87], Portugal [88], Hungary [89], Austria, Spain but also in more temperate regions such as Chzech Republic, Poland, Germany, and the UK [74]. Paulownia may yield profitable economic returns on combined timber and biomass production compared to vine grape in the mediterranean regions depending on subsidies and market prices [22].

The wood is distinguished by a low density and a high extractive content [90]. Depending on the selected *Paulownia* species, the age of the tree and the environmental conditions, the properties of the wood can vary greatly, from the density to the constituents [74, 88]. The reported density ranges from approximately 230 kg/m^3 [89,91] to approximately 295 kg/m^3 [87,92]. Between selections different properties are observed. The low density and hardness of the wood have led to its classification as an inferior wood type for construction.

Therefore one of the researched Paulownia applications is the production of pulp for paper production [86, 87, 90, 93]. Due to the high extract content and low density, the yield of fiber pulp is lower than other wood species [90,93]. While the slenderness ratios are comparable to those of hardwoods [92], the short fibers are likely to result in paper with low strength [86, 90, 92]

Paulownia has also been explored as a raw material to compensate for the expected shortage of other woods or in places with low availability of quality wood [74,76,87,89,94] for wood composite panels such as blockboard [95], or chipboard in different variants from lightweight chipboard [96] to high-density panels made of nanofibrillated cellulose [97]. By adapting production conditions, boards of acceptable quality can be produced [94, 98]. Due to the low wood density, the mechanical strength is low [99], so that a mixture with other types of wood in particleboard [94, 98] or an application in components with low strength such as roof beams [87] is suggested. In the production of blockboards, a weight reduction can be achieved if the low hardness of *Paulownia* is compensated by a veneer layer [95]. The inherently low density is described as advantageous for the production of oriented strand board, where the low density allows for greater densification to increase the strength-to-weight ratio [96].

3.1.3 *Silphium*

The cup plant (*Silphium*), a member of the *asteraceae* family, is closely related to the perennial sunflower (*Heliantheae*) [100]. The perennial nature suggests also a positive influence on soil and ecosystem [101].

The plant is native to the northern parts of the USA and can tolerate colder climates. Cultivars may display frost tolerance to -25 °C [102] but an optimal growth temperature of 20°C is reported [103]. The plant is seed-forming [100], self-pollinating and exhibits a prolonged long flowering period from July to September [101]. The plant exhibits optimal growth on soils with high water availability either due to rainfall, a high groundwater table or a high water storage capacity in the soil [14]. The enhanced drought tolerance observed in this species is attributed to the extended and earlier onset of perennial vegetative growth, which coincides with a period of higher water availability. [14].

The Cup plant is a perennial that has been observed to reach heights of 2–3 m [101, 104]. Its diameter is reported to be approximately 18–20 mm at 10 cm height, 10–14 mm at 130 cm height [104] has the potential to yield 15–25 t ha^{-1} A^{-1} [20,105]. Increasing the nitrogen supply of *Silphium* is increasing the biomass yield [105] but the amounts administered depend on the nitrogen availability in the soil as well as the amount of nutrients contained in the harvested biomass [105, 106].

The harvest is typically performed by forage harvester. In Germany the cultivation area of *Silphium* has recently increased from 400 ha in 2015 to 10.000 ha in 2021, which is mainly intended to be used for biogas applications [107]. The main genotypes used in Germany are currently the seed propagated mixtures consisting of different selections of *Silphium perfoliatum* L . compiled by N.L. Chrestensen Erfurter Samen und Pflanzenanzucht GmbH.

A comparison of the development dynamics of the two genotypes *Silphium perfoliatum* L. and *Silphium Integrifolium* Michx displayed a heterogeneity of the crop [108]. Differences in geographical origins of studied accessions cause disparities in the extent of trait development on the BBCH scale and the timeline of their development [108].

Silphium dry matter can compare with other bioenergy crops [105,109] and can be fermented to biogas [110,111]. Where an early harvest (August) favours carbohydrates, fats and protein required for fodder or bioenergy production [111]. A later harvest in contrast increases the amount of ash and fiber in the feedstock [112] .

Examples for standard applications are such as animal feed [113,114],or as bioenergy substrate [105, 106, 110, 111], where it is sometimes proposed as an alternative to maize [14,115]. In recent research *Silphium* is targeted for material applications in biorefinery processes [19], as biomass source for pulp in paper [20] and as an addition in particle boards [116, 117].

3.2 Biobased Construction Materials

The construction sector is an especially large contributor to both energy consumption and CO_2 emissions [21,118,119]. The development of materials for a bio-based construction materials could reduce the CO_2 footprint of the construction industry. One contribution is to replace fossil-based raw materials with bio-based materials thereby reducing new CO_2 emissions.

In order to facilitate sustainable construction materials based on perennial biomass crops it is germane to identify the relevant steps of product development. The possible interactions between the perennial biomass crops and the performance of the construction material need to be described. Performance and processing differences may persist between biomasses such that the specific amount of CO_2 fixed by application of a biomass in a construction material is individual to its local solution.

The development of the construction materials is focused on the availability of resources and the technical performance of the material. What differences in the suitability of PBC(s) for the production of construction materials exist?

Technical challenges inhibit the material performance by interference between the biomass and the obtained construction material.

The structure to property relations between the biomass and the construction material are thus influenced by the biomass processing. The processing of biomasses to a feedstock for construction materials again requires energy and chemical input and thereby causes its own set of emissions further impacting the CO_2 balance. Therefore it seems appropriate to strive for the lowest processing intensity of the biomass while maintaining satisfactory technical performance to reduce the CO_2 emissions as much as possible. The intricate interaction between necessary processing and technical performance is evaluated for two example construction materials of bio-based light concrete and self-binding fiberboard.

3.2.1 Self-binding Fiberboards

The economic cost distribution for the fiberboard production process splits mainly to approximately 32% for raw materials, 34% for binding agents, the remainder being energy requirements and labor [120]. Wooden particle- or fiberboards are generally produced by chipped or milled wood that is combined with a thermosetting binder to a pressmat and hotpressed either in a dry process for rough chips or in a wet forming process with finely ground material with an added binding agent. Due to the included comminution step homogenous sheet materials can be produced and lower quality resources such as sawdust can be employed above their energetic value. Resource scarcity and competition for woody resources as well as the availability of underutilised lignocellulosic biomass is driving some research interest to board or plate materials formed out agricultural residues [68, 69, 120–123]. However, the reaction of the binders may be specific to chemical environments and may impose problems when used on different biomasses [64, 124]. The binder systems used are mainly urea formaldehyde, melamine urea formaldehyde which are beeing critically assessed due to health concerns for volatile organic decomposition products such as formaldehyde [124, 125]. Other binding agents such as polymeric diphenylmethanediisocyanat (PMDI) are more expensive and not inherently with each biomass [124]. The amount of binder required to achieve boards of sufficient performance, is a relevant economic factor due to the high binder prices [124]. Alternatives to the commonly used binding agents are starch-citric acid binders, plant based protein derivatives such as legumes, surface activation by chemical treatments such as fentons reagent, lignin based binders [126, 127], or processing to self-binding fiberboards [125].

Self-binding boards can be produced by hotpressing of lignocellulosic biomass where the applied heat and pressure cause an adhesive effect between particles [125]. It is supposed that self-binding boards display a benefit in a life cycle assessment. Possible benefits reside in expected biodegradability, reduced formaldehyde emissions [125, 128], and a reduced greenhouse gas potential due to the lack of petrochemical binding agents [125].

The core production process involves a comminution step, a mat forming process, and a heated compression step. Self-binding can be caused if particles are compressed to good contact and sufficient heat is supplied. The supplied heat can function to deform or partially melt the thermoplastic lignin contained in the biomass to cause the adhesive effect [125, 128]. Depending on the precise pressing conditions complex chemical reactions may take place. Biomass constituents including hemicellulose, organic acids and lignin partially degrade and partake in subsequent polymerization reactions [128]. The precise mechanism of bonding is therefore difficult to determine and both process and biomass specific [128].

The choice of the process conditions, and the biomass therefore determines the strength of the formed particle to particle bond and the performance parameters of the produced boards. By adapting the production parameters such as pressure, temperature and pressing time as well as pretreatment methods to increase the surface availability of lignin like steam explosion the performance parameters of the boards can be improved for specific applications [68, 69, 71, 120, 125, 129–132].

Different biomasses tested include different agricultural residues such as banana bunch [133], sugarcane [134], olive- and date prunings [129, 135]. While it is certainly sustainable to use all available residues in the highest valued applications some of the researched biomasses lack the scalability and resource security that are desirable for industrial application.

The perennial biomass crops in this dissertation *Miscanthus* and *Paulownia* have been used in self-binding fiberboards. *Miscanthus* has been employed in different variants after steam explosion pretreatments and has displayed very high performance capabilities [68–71, 121, 136, 137]. *Paulownia* has been ultra micronized to form high density fiberboards from nanocellulose [97, 138].

3.2.2 Vegetal Light Concrete

Concrete is the second most volumetrically used material in the world, surpassed only by water [139]. By mixing cement, gravel, sand, and water concrete is formed as primary building material. Variations of the mixture are also employed as mortar, screed, and as a filling material. Cement undergoes a chemical hydration reaction with water, forming calcium silicate phases which give concrete its strength. The cement is produced by sintering of crushed limestone and specific clays in a kiln to obtain calcium oxide and aluminosilicates. The energy requirement and CO_2 emissions are correspondingly high [140] at approximately 0.6 t CO_2 emitted per ton of cement [141]. Upon contact with water, the resulting cement undergoes an alkaline hydration reaction, resulting in the growth of various crystals that can form a solid substance. In some quarrying sites, the natural limestone compositions already possess the necessary elemental composition, eliminating the need for additional mineral mixtures to obtain a binding cement [142]. The composition of the mineral mixture determines the type of cement produced. Portland cement is the most significant type of cement because it is derived from commonly occurring minerals, which enables widespread production. Concrete properties differ in terms of their final strength, the speed at which solid concrete is formed, the leaching of ions, their resistance to water and moisture, and their resistance to acids [141].

There are multiple methods for reducing the emission rate of concrete building materials. One method for reducing the total amount of emitted CO_2 is through process optimization. This can be achieved by reducing the amount of cement by equivalent functional materials derived from an alternative source, such as recycled concrete or pozzolanic substances like fly ash [143]. Utilization of alternative fuels for kiln heating may reduce the amount of required fossil energy input in cement production [144]. Biomass may partially substitute fossil fuels provided that sufficient heat is generated. This can result in a reduction of emissions from energy consumption [141].

A further effect can be achieved by reducing the transport weight of the individual components and shifting to light concretes with reduced structural strength by substituting sand or gravel with a light aggregate such as fumed silica or biomass [145, 146]. Especially in applications where the high compression strength of concrete is secondary and a high volume is required the substitution of mineral aggregates by light aggregates can display sustainability benefits by decreasing the thermal conductivity.

By using biomass as a light aggregate vegetal concretes are formed, where CO_2 emitted during the production of the cement can be offset by the incorporated biomass. Different types of biomasses or agricultural residues have been tested in different cement mixtures. Biomasses were varied from Spruce strands [147], over alfalfa [148], hemp shives [146,149,150], to *arundo donax* [151], sunflower stalks [152], *Phragmites* [153] and *Miscanthus* [153, 154]. Structural improvement of the cement matrix by means of biomass is difficult to achieve such that benefits of biomass addition are sought in density reduction and its coupled effects [155]. By addition of biomass to lightweight concretes, acoustical- [156–158], and the thermal- insulation of the concrete [118, 157, 158] could be improved by reduction of the overall density.

However, biomass addition is not trivial as it may lead to a reduction in the strength of the concrete [58, 147, 159]. Compatibility issues have been identified between cement and biomass [146,147,160,161]. The degradation of sugar and lignin in an alkaline environment results in their release into the cement, which in turn delays the setting speed and reduces strength development [162].

Furthermore, water uptake by the biomass may interfere with the set water-cement ratio (w/c) [163, 164]. The w/c affects the workability and compactability of the fresh sludge, thereby influencing the final density and strength.

If vegetal concretes are obtained compression strength values for untreated fibers are often reported do be around 1–2 N mm^{-2} [159, 163, 165]. Different studies varied numerous parameters when incorporating biomass into concrete to increase the compatibility [149, 153, 154, 164, 166, 167]. The pretreatments suggested in literature vary in their process complexity and chemical and energetic requirements. Biomass pretreatments are performed to negate detrimental effects such as mineral coatings of the biomass [155] with concrete [57, 60, 164] or with sodium silicates [168].

Plant fibers from beet pulp [164] and Diss [163] were treated by linseed oils to decrease amount of extractable soluble substances and rate of water absorption for the application in vegetal concretes. Khazma et al intended to develop a biodegradable hydrophobic coating for flax shives based on polyethylene glycol and citric acid (PEG-co-CA) with low energy consumption and reduced the water absorption of flax shives to 100% by optimizing the amount of the applied polymer. Alkaline leaching was also used to reduce the amount of soluble hemicelluloses on banana fiber [169], on hemp after rinsing [170] or by acidic silanization treatments to seal the fibers to increase the compression strength [165].

Different kinds of cement were suggested to overcome compatibility issues [171], as well as binder adjustments with acceleration agents such as Calcium chloride [172] or lime [153], and quick setting binders [118, 153].

Thus biomass and concrete may be combined, but different biomasses show variances in performance and biomass and binder interaction is case specific. Identifying specific biomass characteristics that influence concrete setting could aid in optimizing agricultural genotype selection.

3.3 Scope

This dissertation investigates the feasibility of utilizing perennial biomass crops (PBCs) as feedstock for materials, addressing a critical need for sustainable, bio-based alternatives to fossil-fuel-dependent building materials. By means of a literature study on the example biomass *Miscanthus* in materials applications different routes for the higher valued application in two building materials, vegetal concretes and self-binding fiberboards, were described an transferred as basis for experimental material production. *Silphium*, *Miscanthus*, and *Paulownia* were introduced as suitable PBC for scaled cultivation since they are already utilized for bio-energy production. By processing PBCs with commercially available agricultural machinery such as the hammermill, the research identifies critical biomass qualities, such as particle geometry, and processing parameters, such as sieve fractions and substitution rates, that impact feedstock suitability and material performance. The research aims to develop and evaluate two construction materials construction materials: vegetal concrete and self-binding fiberboards.

Given that biomass could be incorporated as an insulating material in cement systems, when compatibility issues between biomass and the binder are mitigated by adding extra water and use of setting accelerated cement, the feasibility of parenchyma rich *Silphium* biomass as light aggregate in insulating light concrete was evaluated. Through experimental testing of different mixture variations with two distinct mineral binding agents this work assesses the compressive strength and thermal insulation of vegetal light concrete by *Silphium* light aggregates as partial substitute for expanded polymer foams. The following research questions were formulated:

- Can aggregates *Silphium*-based offer a viable, sustainable alternative to
  polystyrene in bonded leveling compounds?
- How does the partial substitution of polystyrene balls with (*Silphium*) affect the performance and properties of lightweight aggregate mixtures in different binder systems?

Improving the CO_2 balance of mineral bound construction materials by introduction of biobased aggregates is limited by the footprint of the used binder and by the amount of usable biomass at the performance requirements. Therefore, the contrasting principle of self-binding fiberboards has been investigated. Self-binding fiberboards have been produced from different plants but lack comparability due to process variations, and the energy-intensive nature of most pre-treatment methods. A comparison of *Picea* as control against *Paulownia*, and *Miscanthus* using simplified processing steps with a hammermill was developed. Through experimental testing of different mixture variations, this work assesses the technical performance of these materials in terms of modulus of elasticity, modulus of rupture, reaction to fire and water resistance, analyzing the impacts of biomass type, and processing techniques to address the following questions :

- Does commercially available agricultural machinery, such as a hammermill, achieve sufficient particle size reduction in *Picea*, *Paulownia*, and *Miscanthus* to produce viable feedstock for self-binding fiberboards?
- How does particle size distribution affect the technical perfor mance of self-binding fiberboards for each biomass type?
- How do *Picea*, *Paulownia*, and *Miscanthus* biomasses differ in their suitability and performance as self-binding fiberboards, particularly when particle size distribution is adjusted?

The study contributes to the body of knowledge on PBCs by defining key biomass characteristics and processing parameters that influence the performance of construction materials. Findings from the experiments inform potential substitution levels for conventional materials and offer a pathway for the construction industry to integrate circular economy principles, as well as providing groundwork for future research into the full lifecycle CO_2 reduction potential of bio-based construction materials.

Publications

4. Increase of Miscanthus Cultivation with New Roles in Materials Production–A Review

Outline

Bibliographic Information

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Abstract: Recent changes in the EU green aims can help to overcome economic obstacles in the slow upscaling of *Miscanthus* cultivation. Using *Miscanthus* can permanently fix CO_2 within building materials thereby aiding the EU climate goals with the increased use of regrowing materials, as well as carbon fixation. Economic obstacles in the slow upscaling of *Miscanthus* cultivation are targeted by recent changes in the greening aims in the EU. *Miscanthus* can fulfill a valuable dual function in aiding the EU climate goals by achieving permanent CO_2 fixation within building materials. In contrast to energetic use, persistent applications create stable markets allowing for a reduced risk in the establishment of long term cultured perennial crops. However, the development of different building materials requires an understanding of the combination of the biological and technical aspects. This work presents an overview of the development of the general aspects for the agricultural product *Miscanthus* and the scientifically reported developments of *Miscanthus* used as feedstock in polymers, particle boards, and cementitious materials. While the product performance can be evaluated, the understanding of the influence by the input biomass as a main contributor to the product performance needs to be reinforced to be successful with a goal-oriented development of *Miscanthus* based products. The key feedstock parameters governing the technical performance of the materials are identified and the knowledge gaps are described.

4.1 Introduction

The European Union (EU) has set key targets to reduce the amount of greenhouse gas emissions, to a level of 20% of the 1990 emissions, by the year 2020 and to reduce further to 40% by 2030 [173]. The Cultivation of perennial biomass crops will increase CO_2 sequestration [174]. Since 2018 *Miscanthus* has been included in the so called 'Greening' of the EU (Regulation(EU)2017/2393), which might be advantageous for farmers to cultivate *Miscanthus*.

The genus *Miscanthus* is part of the grass family (*Poaceae)* of the Poaceace with possible cultivation in a wide geographical context, spanning different climate zones from south to north Europe [32, 175, 176]. The perennial species of this genus were originally introduced as ornamental plants [26].

In recent decades, scientific interest and cultivation has increased due to the expected yield potential of the lignocellulosic biomass [26, 175, 177] with respect to the low input requirements in soil and fertilizer use, shifting the focus to the production of lignocellulosic biomass with *Miscanthus* [36, 175, 176, 178–180]. Recent projections predict that *Miscanthus* will be able to contribute 5% to the global energy needs in the 2090s [181].

Eleven million ha of marginal land in the EU have been deemed suitable [182], due to a high tolerance for abiotic stresses [24] as well as water use efficiency C_4 cycle of the plant [183].

The EU has set goals to increase the contribution of bioenergy, to the total energy production, to 20% in 2020 and 32% in 2030, proportionally [173]. The bioenergetic application of *Miscanthus* is often suggested to contribute to the reduction of fossil fuel consumption [32, 175, 184–189]. Next to the replacement of fossil fuels by *Miscanthus* and the carbon fixation in terms of below ground biomass, a non-energetic long-lasting application of the above ground biomass could actively contribute to the reduction of anthropogenic CO_2.

However, the available biomass is currently limited to 19,000 ha in the EU [32] due to high investment costs of the establishment [32] and the uncertainties in market stability [32]. In order to develop a larger sustainable market, higher valued applications should be co-developed together with the bioenergetic usage.

This work aims to review the conversion of *Miscanthus* for materials production in order to facilitate the guided research of higher valued applications, with a focus on building and construction materials. A short description of the plant with a brief coverage of the agronomic processes and valorisation context will be given, followed by the research in higher valued applications such as fiber material in polymer composites, raw materials in the research around particle boards, as well as insulation panels and the research of cementitious materials.

4.2 Agroeconomic Factors of *Miscanthus*

4.2.1 *Miscanthus* Botanical Summary

In south and mid European climates, *Miscanthus sinensis* and *Miscanthus* × *giganteus* genotypes are suggested for biomass production, while *Miscanthus sinensis* is also recommended for northern Europe [176]. However, only *M.* × *giganteus* is currently grown commercially [32]. The *Miscanthus* plant consists of distinguishable morphological and anatomical main components. The rhizomes and roots serve as permanent below ground biomass and the culms and leaves serve as aboveground biomass.

The leaves of *Miscanthus* are typically long, slender, and fibrous, and they contribute to a significant part of the fresh biomass (30%) [190, 191]. Culms display a smooth epidermis under the leave-sheaths and show a lignified sclerenchyma and cortex in the outer ring of the cross-section and less lignified, porous parenchyma in the inner ring [190].

The vegetation period of *Miscanthus* starts in April/May and ends in the autumn (October) [191]. Depending on the genotype and growing conditions, the plant reaches a height of up to 4 m [24, 25] . In autumn, the senescence of the perennial begins. Nutrients and sugars are relocated to the rhizomes; however, the time point of completed senescence and $\geqslant$ 80% loss of greenness may vary between late October to late November [27]. During autumn and over the winter period, genotype dependent leaf losses and drying of the culms occur. Depending on wind and snow conditions, some lodging of the culms could also occur [176].

The chemical composition of *Miscanthus* is reported as 40-60% cellulose, 20-40% hemicellulose, and 10-30% lignin [17] in the organic compounds, and 2-3.5% ash as mineral components [18]. However, the chemical composition is subjected to numerous influences, such as inherent genotypic variations [17,32,39,192], the weather conditions of the growth period, the local soil quality, the agronomic practices in fertilization [32,192] and the harvesting time [32,176,192].

4.2.2 Establishment

The establishment of *Miscanthus* is considered as a main cost factor during the lifespan of cultivation, with estimated costs shares as high as 70% [188]. The main contributor to these estimated costs, next to the land use of the field, are the efforts in rhizome propagation and planting [26, 32, 188, 193, 194]. Furthermore, the initial establishment contributions are in the land preparation before planting, initial fertilizer use, herbicide use and weeding, as well as irrigation during the initial growth period [26,194]. The high prize requirement for the rhizomes is caused by the effort of vegetative multiplication in nursery fields [184,188,194]. Next to the land use for vegetative multiplication, there are fuel and labour costs involved. Rhizome planting is logistically challenging, as live plant parts have to be harvested, transported, and planted without loss of vitality [194]. The technology of *in vitro* propagation is estimated to be even more expensive [32,188,193]. Current efforts have been made in the development of alternative propagation techniques, such as seedling plug planting of greenhouse cultivated plugs [32]. Alternatively, direct sowing under mulch film has shown potential even though the survival rate was only 20% and the agronomic protocols need to be refined for economic applicability [34]. Depending on the soil and climate conditions, the establishment methods have to be adapted. The Solutions includes the planting of rhizomes under a film cover to emulate a warmer, more humid micro-climate [32]. After the establishment of the stand, the cultivar yield is expected to be stabilized after 2–3 years and remain roughly constant during the use phase, up to 25 years [32].

4.2.3 Biomass Harvest

Miscanthus can be harvested in different forms, depending on which kind of aggregate size will be required for the intended application. Some applications, such as thatching [195] require long or full-length stem materials. Other applications, like particle board [61], fiber in polymer [43] composites, or as addition to concrete [56,196], may not require large stem pieces and accept particles as input. In that case, harvesting can be carried out by a maize harvester where the biomass is directly blown onto a transport vehicle. Alternative harvesting and transport modes encompass on-field baling where the integrity of the stems is not a prerequisite [197]. If longer transportation distances are unavoidable or direct energetic applications are intended, harvested material can be further compacted to pellets or briquettes [198].

The harvesting process has major influences on the quality and economics of the biomass and no general applicable standard has been set to date [197, 199–201]. The energy requirements during harvest can be reduced by modifications to the machinery, including oblique angle cutting [202, 203]. An increase in harvesting speed was obtained by using serrated blades to increase the theoretical field capacity from 1.35 to 2.23 ha h^{-1} [204]. Not only the particle size and storage stability are affected but also the biomass composition, in terms of moisture and actual chemical component availability, are directly influenced by the harvesting time and on-field processing [17, 26, 35, 176, 205].

4.2.4 Yields in Geographical Context

Local production of *Miscanthus* appears to be feasible in a European context [26, 175]. Lewandowski et al. [26] reported succesful cultivation of *Miscanthus* in non-irrigated fields of *Miscanthus* × *giganteus* in a wide range of locations, from Portugal to Sweden, and a broad, viable range of climates for cultivation was indicated in the literature [26,32,175,176]. The general yields, over a range of climatic conditions, were put on 10 t ha^{-1} to 25 t ha^{-1} in a European context, by Lewandowski et al. [26]. The yields increase in warmer climates, but decrease in the case of drought stress have to be expected [26, 175, 188].

4.2.5 Transport

A challenge to the production of materials from *Miscanthus* is the availability of biomass at the specific processing site. The biomass demand of a production facility has to be met without exceeding a proportionality of the transportation cost. The transportation proportionality is a pay-off between the biomass value and volume against the transportation distance. The low density and low value material of *Miscanthus* necessitates a local value adding chain to be economical. Assumptions for the viable transport distances of raw *Miscanthus* biomass are generally valued below 100 km [184, 187, 198]. The transportability remains limited by densification options in labour and energy demand, as well as the alteration of material properties instituted by the chosen densification process [32].

4.2.6 Feedstock Parameters

The feedstock parameters can be divided into three main fields: the macroscopic scale, the microscopic scale and the molecular scale. The macroscopic scale consists of demands to the transport qualities, including the bulk density [198, 206], or the culm and chip size [190], moisture content [43,44,176,206] and the aspect ratio [45–47] of the biomass. On the microscopic scale, the surface properties, in terms of roughness [2,45,62], pore sizes [62,63], surface chemistry [47,48,190], and fibrillation of particles [68], could be relevant. For the molecular scale, one may consider the chemical aspects of the feedstock. This would divide the chemical composition in relative amounts of valuable extractable or separable molecules [32,39,207], as well as possible process-inhibiting constituents [57, 176, 183, 208, 209]. The parameters on the molecular level of the feedstock are dominated by the chemical composition and the molecular structure of the extractables, due to the stage of development and genotype.

The growth and senescence results in morphological differences, such as the leaf-stem ratio in varieties or hybrids, thereby alter the composition of the feedstock and the viability for different applications, like fermentation [210] or direct combustion [35, 211, 212]. Abiotic stresses influence the growth and senescence cycles, such that varieties may be screened for optimum climate-plant combinations [32, 35, 176]. Furthermore, variations in the extracted molecules from different plant parts may be expected. Lignin extracted from different plant components (stem and leaf) displayed different polymer characteristics [39]. Additionally, the chemical structure of the biomass can be relevant in terms of the embedding and possible release paths of molecules in the biologic matrix (i.e., recalcitrance and crystallinity) [32, 41, 183, 207, 213]. Kaack et al. [213], for example, related chemical and morphological traits. Statistical analysis yielded a statistical relation between the modulus of elasticity, area of outer ring, and lignin, as well as the area of parenchyma and cellulose, and the area of vascular bundles and cellulose, thereby suggesting , *Miscanthus* × *giganteus* as part of a likely group of genotypes for the production of building materials. Research and knowledge are thus highly specific and dependent on the chosen application.

The agronomic biomass end-product *Miscanthus* will have to be converted to a viable feedstock depending on the chosen valorisation chain. The applications are diverse, such that feedstock requirements depend on the follow up refinement. An application such as light weight concrete, as examined by Pude et al. [154], requires stem fragments with relevant parameters on the macroscopic scale; whereas the fabrication of nano-cellulose as a polymer reinforcement, as investigated by El Achaby et al. [214], requires a feedstock rich in crystalline cellulose.

Energetic applications are well understood such that the feedstock characteristics can be defined by the parameters of processing, pre-treatment, transport, storage and harvest, and the chemical composition of the plant variety itself [17, 32, 207]. The research is focussed on the latter plant specific traits, such as increased biomass yield [32], reduced recalcitrance (lignin content) [186], and increasing sugar or starch content [188, 207], but the genetic viability required for successful mass-scale deployment was determined to be a key issue [180]. On the other hand, in the context of biorefinery the pre-treatment parameters and methods structurally researched are aiming at less energy intensive [17] or less chemically demanding processes [17, 187]. A recent research model is even targeted at the online analysis of a feedstock in order to be able to adapt the running process by the input feed [215].

The combination of optimal feedstock parameters is complex as components, such as lignin, may be used as value generating sidestream, depending on the used processes [39, 216].

4.3 Polymer Composites

4.3.1 *Miscanthus* Biomass in Polymers

Modern polymeric materials with natural fibers in composites have gained much attention as reinforcements [50]. The function of the added material can vary from the reduction of weight or price of a composite [44, 46, 49] up to the improvement of structural parameters of the product [46, 217]. However, the employment of *Miscanthus* biomass in polymer systems is complex and an overview on the general aspects and tendencies is given.

Difficulties in the reinforcement mechanisms are caused by the fiber to matrix compatibility [47], the dispersion of the fibers within the matrix [47], and possible degradation of fiber during the processing [43,44,51]. The fiber to matrix bond may be strongly influenced by hydrophilic particles and hydrophobic polymers, leading to insufficient adhesion [45,47,48,217]. Matrix compatibilizers can be used to improve the interface adhesion by blending the polymer with a co-polymer, such as maleic anhydride [45,47,53], grafted polypropylene, or by reactive activation with a peroxide [52]. However, the necessity for compatibilization is dependent on the specific polymer system as a good interfacial bond was reported in poly(3-hydroxybutyrate-co-3-hydroxyvalerate) (PHBV) [48, 54] .

The biomass particle parameters of size, alignment, and dispersion in the matrix determine the degree of mechanical reinforcement that is possible [45, 47, 51, 52]. A strong influence of fine particles *via* the reduction of mechanical properties and the necessity of the removal of irregular dust was reported by Girones et al. [45]. A homogeneous dispersion of fibers is required as reinforcement effects are anisotropic and the misalignment of fibers can lead to a weaker specimen [46]. Smaller fibers with a high length to width aspect ratio are related to tensile strength increases, due to the higher specific surface area [45]. The processing of polymers and fibers can degrade the integrity of the fibers by process heat and prolonged heat (residence time) [44–46], as well as by physical shear forces causing fiber size reduction [46, 51]. Gamon et al. [51] have investigated the influence of different extrusion screw speeds. They reported that extrusion at 150 rpm was the best trade-off between low energy requirements for slow speeds and sufficient homogenisation and fiber bundle preservation at high speeds, in a twin screw extruder. Other pathways of *Miscanthus* biomass incorporation into a composite were tried with alternative pre-treatment methods, such as pyrolysis [55, 218], corona discharge [219], and the extraction of cellulose crystals [214, 220].

4.3.2 Technical Aspects of Composite Manufacturing

Johnson et al. [43,44] investigated the influence of *Miscanthus* fiber loading, particle size, and the processing temperature on the impact performance of biodegradable Novamont Mater-Bi© Polymer. The processing variables were the extrusion speed (15%, 30%), fiber loadings (10%, 20%), particle sizes ($\geqslant$ 3 mm, $\leqslant$ 1 mm), and temperatures (175℃ , 190℃). The impact load of the fiber reinforced compound was increased up to 30% for increased heat and a 20% fiber loading of partilces below 1 mm. The increase in impact load was assumed to be caused by weak adhesion between the *Miscanthus* fibers and the polymer-matrix, and resulting in fiber pull-out. Temperature was assumed to be a main factor and was later validated as the only significant factor for these process conditions [43, 44, 221].

Kirwan et al. [46] researched the influence of different processing parameters on the flexural rigidity and modulus of *Miscanthus* in a commercial poly(vinyl alcohol) (PVA) blend. Analysis of the parameters was carried out by a fractional-factorial design of experimental statistical method, in order to find the influence of the fiber length (2 mm, 4 mm), volume (10%, 20%), washing temperature (72 h at 25 ℃, 3 h at 100 ℃), processing temperature (190 ℃, 200 ℃), and the blending of poly(vinyl acetate) PVAc (5%, 10%) into the PVA. The flexural rigidity was improved from 61.8 to 123.4 N mm^{-2} with a combination of a hot washing with short fibers at 20% fiber load in a 5% PVAc /PVAXX W63 blend processed at 200 ℃ . The same combination reached an improvement of the flexural modulus from 2700 to $\approx$ 9500 N mm^{-2}. Combinations analysis of different factors showed that temperature was the main parameter and this is in accordance with other reports [43,44,221]. A loss of interfacial adhesion between particle and the matrix was observed by scanning electron microscopy (SEM). Furthermore, the hot washing fiber pre-treatment was concluded to be beneficial to the modulus and strength. The removal of soluble starches were assumed to have caused increased adhesion leading and strength. The analysis of the fiber length was impeded by size reduction during the processing and analysis of reclaimed fibers in size and aspect ratios of the longer feedstock were found to be similar to the short fibers.

Bourmaud et al. [47] analysed the mechanical properties at different fiber loading ratios (20%, 30%, 40%) with maleic anhydride (MA) compatibilizer (0%, 2%, 5 %) in two different matrices of polypropylene (PP) and polylactic acid (PLLA). In the PP matrix, the addition of 2% compatibilizer improved the tensile modulus from 1051 to 2309 N mm^{-2}, 2742 N mm^{-2}, and 3453 N mm^{-2} for increasing fiber loadings. In the PLLA matrix addition of 2% compatibilizer improved the tensile modulus from 3401 to 5337 N mm^{-2}, 5492 N mm^{-2}, and 6652 N mm^{-2} for increasing fiber loadings. The improvements of the tensile modulus is assumed to be caused by improved fiber dispersion and interfacial bonds. Further addition of compatibilizer did not show significant improvements. Therefore it was concluded that an optimal amount is below the 5% compatibilizer addition. The strength at yield in PLLA decreases from 60.4 to 40.8 N mm^{-2}, 37.9 N mm^{-2}, and 39.3 N mm^{-2} with addition of the fiberss which was attributed to poor lengths and aspect ratios.

Nagarajan et al. [48] incorporated *Miscanthus* at a loading of 30% in a biodegradable matrix blend. The matrix blend used was based on poly(3-hydroxybutyrate-co-3-hydroxyvalerate) (PHBV) and poly(butylene adipate-co-terephthalate) (PBAT). The impact strength of the *Miscanthus* composites was decreased from 369.93 to 35.76 N mm^{-2} while the tensile strength was increased from 21.60 to 23.30 N mm^{-2}. It was assumed that the high lignin content in *Miscanthus* led to increased interfacial bonding with the polymer. Energy dissipation on impact by fiber pull-out was reduced and critical failure of the fibers caused a reduction of the impact strength.

Zhang et al. [54] investigated the feasibility of a co-injected PHBV/*Miscanthus* core in a poly (butylene succinate) PBS/PBAT blend skin. Co-injection molding of a PHBV/*Miscanthus* core into a neat PBS skin decreased the notched impact strength from 29.8 J m^{-1} for a single injected PHBV/*Miscanthus* matrix to 29.2 J m^{-1}. Co-injection moulding of a PHBV/*Miscanthus* core into a PBS/PBAT skin increased the notched impact strength to 139.8 J m^{-1}. The unnotched impact strengths were increased from 112.6 to 322.1 J m^{-1} for neat PBS and 398.7 J m^{-1} for a PBS/PBAT blend. The tensile strength values were $\approx$ 18 N mm^{-2} for a single injected matrix, 28 N mm^{-2} for PBS skinned composite, and 19 N mm^{-2} for the PBS/PBAT composite. The flexural strength values were $\approx$ 32 N mm^{-2} for a single injected matrix, 37 N mm^{-2} for a PBS skinned composite, and 20 N mm^{-2} for the PBS/PBAT composite. The different combinations of the mechanical values led to the conclusion that co-injection molding may be used to design *Miscanthus* composite materials according to specific performance requirements.

Chupin et al. [49] contrasted the common use of culms and tested the reinforcement capabilities of rhizome biomass of *Miscanthus* $\times$ *giganteus* in poly ethylene (PE) composites. Using a fiber loading of 30% with particles retained between 100 μm and 200 μm, sievescreens specimens, for rhizome mass and stems, were produced. The tensile strength decreased for the rhizome composite from 7.62 N mm^{-2} for neat PE to 7.45 N mm^{-2}, while the stem composite increased the value to 13.5 N mm^{-2}. The low tensile strength of the rhizome composite was assumed to be caused by a low aspect ratio of rhizome fragments as well as a lower intrinsic strength due to a higher proportion of hemicelluloses in the rhizomes (35%) compared to the stem (21%).

El Achaby et al. [214] investigated the potential of *Miscanthus* fibers for the production of reinforcing cellulose nanocrystals (CNC) for the potential usability in polymer systems. By employing sulphuric acid hydrolysis on *Miscanthus* $\times$ *giganteus* CNC with an aspect ratio of 37 and a crystallinity of 76% could be obtained. The addition of 8% CNC improved the tensile properties of starch based nano-composite films to 118% in tensile strength and 150% in Young´s modulus.

Miscanthus has been tested in different polymer systems with a strong focus on processing and feasibility within a given combination, as shown in table 4.1.

4.3.3 Summary and Conclusions: *Miscanthus* Polymer Composites

The polymer to fiber bond of *Miscanthus* in polymer systems can be improved by compatibilizing agents blended into the polymer matrix [45, 47, 52, 53]. A high hemicellulose content of the feedstock is suspected of detrimental effects [47, 49]. The main reported parameters governing the per-

Table 4.1: The table displays the different biocomposites produced based on *Miscanthus* fibers/ particles.

Process	Polymer blend	Compatibilizer	Variable/Question	Author/Group
Injection molding	Mater Bi©	-	process variables	Johnson et al. [43]
Injection molding	Mater Bi©	-	process variables	Johnson et al. [44]
Injection molding	PVA	-	process variables	Kirwan et al. [46]
Injection molding	PP	MA	fiber loading / compatibilizer	Bourmaud et al. [47]
Injection molding	PLLA	MA	fiber loading / compatibilizer	Bourmaud et al. [47]
Injection molding	PHBV / PBAT	-	biomasses	Nagarajan et al. [48]
Injection molding	PP	MA	process variables	Girones et al. [45]
Injection molding	PP	MA	genotypes	Girones et al. [45]
Injection molding	PHBV	DCP	feasibility	Muthuraj et al. [52]
Injection molding	PBS / PBAT	MA	feasibility	Muthuraj et al. [53]
Injection molding	PE	MA	feasibility	Chupin et al. [49]
Injection molding	Nylon	pyrolysis	process variables	Ogunsona et al. [55]
Injection molding	PBS / PBAT	-	process variables	Muthuraj et al. [222]
Co-Injection molding	PBS / PHBV	-	feasibility	Zhang et al. [54]
Thermo-compression	PP + PLA	corona discharge	pre-treatment	Ragoubi et al. [219]

formance of the polymer compounds are the high length-to-width aspect ratio of the particles [45,49], the degree of fiber loading [47], and the dispersion of fibers within the matrix [47, 51]. Extrusion of polymers and fibers causes degradation of the biomass under certain conditions [44–46, 51]. In general, a potential for the application of *Miscanthus* as a feedstock material in polymer systems is expected [44, 46, 217]. Further research should include the influences of the chemical composition of the feedstock on the mechanical performance. However, a focus should be on the comminution, the fiber degradation during extrusion, and the dispersion of the particles inside the produced matrix.

4.4 Particleboards

4.4.1 *Miscanthus* in Conventional Particleboards

Currently, several researchers have investigated the possibility of wood substitution with alternative resources and processes [61–64, 66, 125, 136, 223]. In this case, the main driving force is the relatively high competition of the wood resource and the subsequent economic benefit of replacement or partial replacement of wood with existing agricultural by-products. *Miscanthus* is part of the tested alternatives even though it is not strictly a by-product, as the other tested materials are.

Miscanthus may be employed as a wood substitute if a sufficient amount of appropriate adhesive binder is used [61, 64–66]. However, the binder systems were found to be limited by the chemical environment, in terms of an increased pH and pH buffering capacity excluding urea formaldehyde and phenol formaldehyde binder systems [64], as well as a limited surface adhesion on the stalks [62]. Therefore, it was suggested that the chemical environment and surface properties may be modified by high pressure refinement [64]. A full substitution by non-wood biomass was only applicable with *Miscanthus* and at the drawback of increased resin requirements of methylene diphenyl diisocyanate (MDI) for acceptable panel properties [64]. Further drawbacks were expected in the amount of extractives and mineral content in terms of silica [64]. Tool wear by abrasion and chemical corrosion is caused by extractives and silica in the long run [224].

Balducci et al. [63] investigated board production based on parenchyma rich raw materials in order to decrease the compound weight. It was supposed that inherently lighter materials could achieve higher mechanical properties compared to wood particle boards, which are not conform to EN 312 at low densities. The particle boards derived by low density materials reached modulus of elasticity (MOE) values ranging 560-1270 N mm^{-2} and did not conform to EN 312 by MOE (1600 N mm^{-2}) or by flexural strength (13.0 N mm^{-2}) and are thus not directly applicable for general indoor use. The special structure of the *Miscanthus* parenchyma limits the internal bond strength (IB) as the weak link in the superstructure, even though other mechanical parameters indicate the general application of wood substituted boards under dry conditions [65]. In a substitution experiment of wood at two MDI resination levels (4% and 6%), the MOE, modulus of rupture (MOR), IB and thickness swelling (TS) were determined according to EN 312 P1. However, stagnation of MOR (14 N mm^{-2}), MOE (1700 N mm^{-2}) and IB (0.35 N mm^{-2}) was reported for *Miscanthus* with increasing binder content, whereas the wood reference did increase in IB from 0.85 N mm^{-2} to 1.1 N mm^{-2} implying that the particle to binder interaction reached a particle inherent limit in *Miscanthus*. A performed micro-structural analysis showed collapsed parenchyma cells in the *Mis-*

canthus specimen after the IB test. It was concluded that the structure of the parenchyma is responsible for the weak bonds and subsequently suggested that a removal of the parenchyma fraction prior to pressing could improve the board properties.

Tröger et al. [61, 66] investigated the improvement options of wood substituted *Miscanthus* boards by means of flax fiber mat reinforcements [66] and multilayered board constitutions [61]. Flax fiber mat reinforcement of the *Miscanthus* MDI particle boards led to MOE values of 5990 N mm^{-2} and flexural strength values of 39.6 N mm^{-2}, which is comparable to glass fiber reinforced panels [66]. The TS of the *Miscanthus* based -flax fiber reinforced- boards over 24 h was reported as improved by 9.5%, which is half of the used spruce references (15.8-18.0%). However, the internal bond strength (IB) of the *Miscanthus* based board was lowered (1.21 N mm^{-2}) compared to the spruce reference (1.52 N mm^{-2}) indicating that the *Miscanthus* interaction with the binder is not optimal or that the parenchyma introduced a breaking point. Multi-layered systems with 50% *Miscanthus*/wood core and varying surface layers displayed increased bending strength (23.0 to 39.6 N mm^{-2}) and MOE (3500 to 6000 N mm^{-2} by flax fiber reinforcement). However, even the non-reinforced multilayer variant of pure *Miscanthus* strands in the surface layers and displayed performance, which was equal to the reference wood standard produced, suggesting that a partial wood substitution is technically feasible. However, due to the MDI requirement, the economic feasibility was considered insufficient.

At a partial wood substitution level of 50% in Douglas-fir (Pseudotsuga menziesii) boards, PF binders were found to keep the IB at suitable levels [62]. While the parenchyma content likely decreased the IB of the boards, [63, 65], Park et al. [62] considered the slender particle geometry of *Miscanthus* as responsible for the increased MOE at higher wood substitution levels and lower binder amounts (9% vs. 11% PF). An offsetting effect is observed at increased resin content (11%) where the MOE is raised from pure *Miscanthus*: $\approx$ 1600 to $\approx$ 2100 N mm^{-2} for full wood, composition [62]. An improved particle-binder bond was suggested for the wood due to the low porosity and stalk surface properties of the *Miscanthus* epidermis.

4.4.2 *Miscanthus* Biomass in Binderless Fiberboards

A different development in the particleboard research is the investigation of binding processes without the addition of synthetic binders. The two main reasons behind this are the strict legislation on formaldehyde emissions, which poses new requirements on the existing binders, and the high contribution of the binder price to the total cost of materials [223].

Lignocellulosic biomass can be converted into self-adhesive boards. The plastification of thermoplastic lignin is intended to be the main contributor to the binding effect. The binding effects are influenced by mechanical contact, molecular contact, and interaction, chemical bonding as well as the structural integrity of the composite as is elaborated by Hubbe et al. [125] in more detail. Due to the complexity and mutual influence of the effects governing the overall properties of the produced board, a main focus in the researches is the influence of the biomass pre-traetment and processing conditions on the mechanical properties of the board. An overwiev of Miscanhtus based particle boards with and without added binders is given in 4.3.

Velásquez et al. [136] produced binderless fiberboards from material of *Miscanthus sinensis* by steam explosion pre-treatment in a masonite process and analysed the pre-treatment and pressing conditions. The highest total MOE and MOR values reached were 6050 N mm^{-2} and 48.2 N mm^{-2}, respectively, with an IB of 1.2 N mm^{-2}. The feedstock was obtained by severe (4.0) steam explosion treatment at 216 ℃ for 3.5 min and moderate pressing conditions at 180 ℃. By variations of combinations, different estimated response surface diagrams were produced to predict the behavior of a mechanical product parameter by variations in either pre-treatment or processing conditions. The pre-treatment stage was concluded to have a higher influence on the physiochemical properties than the hot pressing stage. Next to the physical particle disintegration, chemical degradations also occur during the masonite process. Improved water absorption and thickness swelling behavior was assumed to be correlated with decreased hemicellulose content by hydrolysis for increasing the pre-treatment severity. The increased physical degradation also led to an improvement of IB, by increasing particle contact. In contrast to the *Miscanthus* particle boards produced with binders, the binderless boards have a higher density by compression and physical disintegration, such that the porous structure of the parenchyma would be collapsed, and water absorption takes place through swelling of the hydrophilic fibers.

Velásquez et al. [121] researched the physio-mechanical response of binderless *Miscanthus* fiberboard through size reduction of a steam exploded raw material through grinding. The IB was improved by 50% *via* grinding. It was concluded that grinding size reduction of particles treated with low severity parameters led to defibrillation of fiber bundles and increased the contact area and the strength of the bonds in pressing. Due to uninhibited MOE and MOR values, a significant length reduction of the fibers was considered improbable.

Velásquez et al. continued with [137], the experimental influence of feedstock and process conditions by investigating an increased span of parameter ranges in pre-treatment severity and pressing, and the subsequent changes in mechanical particle board parameters. The specific conditions are evaluated to maximise the MOE and MOR values, which reached theoretical values of 7500 N mm^{-2} and 61.2 N mm^{-2}, respectively. In the steam explosion pre-treatment, a temperature of 203 ℃ with a pre-treatment time of 7.35 min is found to be optimal and pressing in a three stage process at 220 ℃ and a pressure of 12.1 MPa is considered optimal.

Next to the pure binderless boards, Velásquez et al. [136] investigated the addition of exogen kraft lignin powder for partial substitution of *Miscanthus sinensis* by steam explosion pulp in hot pressed particleboards. By pressing with reduced temperatures at 170 ℃ with 20% exogen lignin substitution, MOE values of 5900 N mm^{-2} were predicted. Further increasing of the temperatures and lignin content was reported to cause internal bubbles, limiting the bonding of the composite. It was concluded that substitution of the pulp is possible without quality loss if pressing temperatures are reduced. Further improvement was reported for the mixing of fibers with lignin prior to the steam explosion. Two causes were suspected for the improvement, first, the removal of volatile substances that otherwise may lead to destabilizing formation of gas bubbles, and second, an increased homogenisation of the exogen lignin and cellulosic fiber. Reduced material requirements by improved properties were concluded to translate into economic benefits with reduced energy requirements of the process. The volatile substances suspected as cause for bubble formation at elevated temperatures should be removed during the steam explosion and better homogenisation of exogen lignin and cellulosic fibers is likely. Being able to reduce the amount of steam exploded material and the required pressing temperature translates into economic benefits in terms of energy.

Binderless boards without steam explosion pre-treatment were produced by Moll et al. (2018) [225] by hot pressing hammer-milled *Miscanthus* × *giganteus* particles. The particle boards were produced from different sieve-fractions of a single hammer-milling step, in order to determine the influence of the particle size on the mechanical board properties. The particle fraction passing a 0.25 mm sieve-screen resulted in the highest MOE (1200 N mm^{-2}), whereas particles between a 0.5 mm and 0.75 sieve-screen resulted in much reduced MOE values (190 N mm^{-2}). It was concluded that a good control of the fine fraction would be required to produce particle boards in hot pressing with specific target properties.

4.4.3 *Miscanthus* Biomass in Insulation Panels

Due to the parenchyma content of *Miscanthus* and the accompanying increased porosity compared to woody biomass, inherent thermal insulation properties are postulated [1,154]. In this context, insulating particleboards have been evaluated for different technical aspects.

El Hage et al. [67] evaluated the potential of a flame retarding chitosan binder for a *Miscanthus* and recycled textile fiber biocomposite. The chitosan binder was developed with different aluminum trihydroxide filler contents while the biocomposite composition was varied in *Miscanthus* to the textile fiber ratio. The thermal conductivities were reported to vary between 69-90 mW*m^{-1} K^{-1} where a higher textile content (0-100%) increased the thermal conductivity by increasing density and decreasing the porosity. Despite higher densities (250 kg m^{-3}) compared to conventional insulation materials and slightly increased thermal conductivity values (λ values) the chitosan bound biocomposites were still considered as insulation material. The fire rating Euroclass E could be obtained by a combination of chitosan improved by addition of anorganic filler. The authors therefore concluded that there was a potential of the flame retarding insulation material. The fire behavior of chitosan was reported as interesting and further improved by anorganic filler integration, such that the Euroclass E fire rating is obtained, and an overall potential of the system was concluded.

Eschenhagen et al. [1] produced and compared insulation panels from sunflower stalks and *Miscanthus* × *giganteus* by testing different natural based binder mixtures. The mechanical values were tested in Youngś modulus and Youngś bending modulus, and the thermal insulation properties were evaluated. The binder systems tested spanned binder to water ratios from 10-40%, and three binders based on starch, casein, and gelatin. Due to the low integrity of the formed panels, only two binder systems with *Miscanthus* (starch, casein) were used in structural testing and three for the sunflower compounds. Further integrity problems limited the tested boards. From the *Miscanthus* two starch based boards (20%, 30%) and two casein based boards (30%, 40%) were tested. The sunflower stalk boards were tested with starch (20%), casein (20%, 30%, 40%) and gelatine based binders (30%). The thermal conductivity and thermal resistance were compared for 20% binder systems in the *Miscanthus* /starch and sunflower/casein system as shown in table 4.2 .

Table 4.2: Thermal conductivity λ and thermal resistance R according to temperature T for composites panels with 20% binder content [1].

	Miscanthus /starch (20%)		Sunflower/casein (20%)	
T (°C)	λ (mW m^{-1} K^{-1})	R (m^2 K W^{-1})	λ (mW m^{-1} K^{-1})	R (m^2 K W^{-1})
10	57.02	1.070	65.24	0.680
25	61.27	0.992	70.42	0.631
40	67.55	0.900	77.42	0.573

Note. Reprinted from " Investigation of Miscanthus and Sunflower Stalk Fiber-Reinforced Composites for Insulation Applications" by Arne Eschenhagen et al., Advances in Civil Engineering, Volume 2019, p 6

Based on the combination of mechanical and thermal properties, a feasibility of *Miscanthus* based insulation boards is concluded to be possible; however, characterizations of the durability in physical and biological aspects, as well as further determination of the standard insulation properties are suggested.

4.4.4 Particleboards: Summary

For the production of particle boards, both adhesive and binderless systems are possible. However, the binding agent needs to be compatible with the chemical environment given by the *Miscanthus* feedstock, as well as the chosen processing conditions. In a binderless system the technical performance is strongly influenced by both the process of board production as well as the pre-treatment methods and comminution of the biomass. While the influence of the production parameters is quantified, the physical and chemical parameters, leading to improved board performance, have not been quantified yet. Several factors influencing the bond strength reside

Table 4.3: An overview of different projects describing *Miscanthus* based board or insulation materials.

Process/Layer	Added Binder	Pre-treatments	Question/ Variable	Author
Multilayer	MDI	-	Feasibility	Tröger et al. [61,66]
Single layer	PF	-	Process variables	Park et al. [62]
Single layer	MDI	-	Feasibility	Balducci et al. [63]
Single layer	MDI	-	Process variables	Klimek et al. [65]
Hot press	-	Steam Explosion	Process variables	Velásquez et al. [121,137]
Hot Press	Exogen Lignin	Steam Explosion	Feasibility	Velásquez et al. [136]
Hot press	-	-	Process variables	Moll et al. [225]
Single layer	Chitosan	-	Feasibility	El Hage et al. [67]
Single layer	starch/casein/gelatin	-	Feasibility	Eschenhagen et al. [1]

in the surface properties of the particles and the low structural integrity of the parenchyma. A separation of the parenchyma and an analysis of the comminution method on the mechanical performance may be advisable. Differences in the chemical composition and morphology of various *Miscanthus* genotypes may be expected to cause variations in mechanical parameters under similar processing conditions.

4.5 Concrete Systems

4.5.1 *Miscanthus* Based Concrete

Concrete production is responsible for around 6% of the global annual CO_2 emissions. Addition of biomass into concrete would reduce CO_2 generation [226] and sequester carbon in building materials. With political development in the European Union, the thermal conductivity of concrete needs to be improved, in order to reduce the energy demand in buildings. The reduction of energy demand for concrete can also be affected by the reduction of energy demanding resources, such a s steel or glass fiber in concrete [2]. *Miscanthus* fibers have a tensile strength of 373 N mm^{-2} [227] and a compressive strength of 56.9 N mm^{-2} [227] and could act as reinforcement, if a good fiber to matrix bond can be established [2, 57, 154]. Furthermore, *Miscanthus* biomass is silicate rich, as 74% of the ash is composed of silica [228] and the structure of porous parenchyma, which are both considered to pose potential benefits in a cementitious matrix [190]. The parenchyma content and the concordant pore structure are expected to reduce the thermal conductivity of the concrete [154, 190, 229]. Insertion of *Miscanthus* into concrete reduces the density of the composite material through addition of the lighter biomass. Both effects of density reduction and pore introduction are common to both thermal and acoustic insulation materials such that improved acoustic insulation properties are expected [56]. Employment of *Miscanthus* in concrete is thus researched for several applications, such as porous admixtures [56, 190, 196], as reinforcement [2, 60], and as alternatives to other bioadmixtures [57].

4.5.2 *Miscanthus* Compatibility with Concrete

Portland cement is the most common type and also the main type of binder considered in the different studies. The following section will cover the common compatibility issues between biomass and Portland cement. The compatibility of the binder and the biomass should have a key role in the mechanical properties of the produced concrete. The setting reaction of Portland cement is a sequence of crystalline systems that hydrate and redevelop [230]. By the amount of water available for hydration the equilibrium reactions of the respective crystal phases are determined [230]. Excess water may induce phase separations and layer formation, and excessive pores in the concrete [231]. Water shortages reduces the flowability of concrete and thus limit the compaction [232]. The water to cement ratio of the mixture thus becomes a relevant parameter for the overall properties of the matrix. Biomass can both absorb and release water, thereby changing the local water to cement ratio [59]. *Miscanthus* has a high water absorption capacity between 200 and 600% depending on the comminution method and particle size [60, 233], and can thus affect the water to cement ratio. The time for water uptake of *Miscanthus* fibers was found to be in the short minute interval [60, 154] such that water deprivation effects take place during the early hydration phase of the concrete. Direct treatment the pre-soaking of *Miscanthus* fibers [56, 60] or the addition of water to the concrete mixture have been suggested [56, 59, 60].

Further arising problems include particle swelling with water uptake and shrinking with drying, or particle degradation in alkaline media, which may leave excessive voids and decouple fibers from the cement binder [2, 59, 60]. Different strategies to limit the water absorption have been attempted. Physical processes that densify the fiber and close pores, include the hornification by cycled wetting and forced drying of *Miscanthus* [60] or the encapsulation of *Miscanthus* by mineral or organic agents that seal the particle by a non-permeable or hydrophobic layer [56, 60].

Organic extractives can delay or inhibit the setting process and ultimately decrease the mechanical properties of the formed concrete [59]. Influencing biomass constituents such as sugars, lignins, and organic acids are suspected to complexate calcium ions and thus slow the equilibrium formation by competition, or disturb the equilibrium by adsorption [57]. Equal inhibiting effects may be caused by absorption of Ca^{2+} in the biomass. Different pre-treatments to leach the available organic constituents of *Miscanthus* have been carried out. The range spans extractions with hot water [60], as well as leaching and silanization of *Miscanthus* in different chemical environments [59], to the use of saccharification residues with a supposedly reduced amount of extractable sugars [57]. Portland cement forms an alkaline environment, when the hydration reaction occurs [230]. Biomasses can buffer the pH changes by ion exchange capacities, release/reaction of organic acids, or by a degradation reaction with an alkaline solution and the subsequent release of fragmentation products [58].

4.5.3 Various *Miscanthus* Particle Influences on Concrete Properties

Pude et al. [196] produced concrete samples from chopped culm pieces with a water to cement (w/c) ratio of 0.8 to determine the influence of the used genotype on pressure stability. Four different genotypes were used: *M. × giganteus*, *M. sacchariflorus*, *M. sinensis* and *M. 'Robustus'* and they displayed variations in pressure stability, depending on the genotype and harvesting year. *M. sinensis* and *M. 'Robustus'* showed the lowest compressibility values (0.41 and 0.28 Nmm^{-2}, respectively) and were hence concluded as inadequate for cement applications. A strong influence of the batch is displayed in the pressure stability differences between the two years. The varieties *M. sacchariflorus*, *M. sinensis* and *M.'Robustus'* displayed a stability reduction to roughly 25% of the 2001 values while *M. × giganteus* did not vary, essentially (0.74 and 0.75 Nmm^{-2}). Growing period related quality parameters influenced by the year's climate or plant stresses thus have a strong influence in the pressure stability of concrete and need to be identified [196]. The water to concrete ratio applied in this study is significantly higher than the suggested ratios for Portland cement (0.5) and may have contributed to the low strength values [60]. The high water uptake of the culm pieces is described by Pude et al. as beneficial to the strength due to saturation with concrete sludge.

Acikel et al. [2] attempted to reinforce concrete of different cement dosages with a w/c ratio of 0.5 by varying the biomass input ratios and different comminution styles. The fibers were ground, cut, embedded as reinforcement, and introduced as mixture. An effect of the biomass loading, the comminution method, and the concrete dosage, was observed in strength of compression, splitting, and flexural strength, as shown in table 4.4. Fiber loading of grround *Miscanthus* (2% and 4%) improved the strength compared to the reference samples. Increasing the cement dosages displayed higher base values. The increase for compressive strength is reported as 9% to 25%. The strength increase at a high cement dosage with increasing fiber load appears to reach a limiting value at high cement dosage of 46 N mm^{-2}, as shown in the table 4.4. The splitting and bending strength are reported to increase by 4% to 8%, where the bending strength also appears to reach a final value, as can be seen in table 4.4. For the cut fibers, the strength parameters did display a reduction that was explained by the smooth plant surface and an insufficient bond to the concrete matrix.

Table 4.4: Adaption of selected mechanical properties of *Miscanthus* concrete specimen by Acikel et al. [2]

Cement dosage [kgm^{-3}]	Grinded fiber loading	Compression strength [N mm^{-2}]	Splitting strenght [N mm^{-2}]	Bending strength [N mm^{-2}]
300	0%	35	2.7	5.5
300	2%	37	3.0	5.8
300	4%	38	3.3	5.9
350	0%	36	2.9	5.9
350	2%	37	3.3	6.1
350	4%	38	3.6	6.2
400	0%	36	3.2	6.2
400	2%	46	3.5	6.8
400	4%	46	3.9	6.7

Furthermore, crystallization effects were observed on the smooth surface of the biomass indicating, a phase separation [2].

Le Ngoc et al. [57] employed *Miscanthus* residues (*Miscanthus* × *giganteus*, at harvesting time in November, during the second year of cultivation) of two different saccharification pre-treatments in cement mortar with w/c of 0.46. The two biomass pre-treatments were dilute heated sulphuric acid and an aqueous ammonia treatment intended to reduce the inhibitory effects caused by lignocellulosic biomass in cement. The biomass was added as a water saturated mass and displayed an increased outflow speed in the composites, from 3s28 s in neat cement paste to 1s94-2s5 s for the residues and raw *Miscanthus*. However, the polysaccharides and lignin contents of both pretreated biomasses were found to be increased,and the setting of the concrete was delayed. The effect could be limited by the addition of CaCl employed as a setting accelerator.

While the flexural strength appeared unaltered ($\approx$ 3.0 N mm^{-2}), the compressive strength was reported to decrease. The reduction is 85% against cement ($\approx$ 66 N mm^{-2}) and by 62% against raw *Miscanthus* cement specimen. The decrease is explained by an increase in the pores of the mineral matrix. Another explanation is a possible modification of the calcium silicate hydrate (C-S-H) crystal phase by the formation of salts between calcium and the organic constituents. Despite of the reduction of mechanical properties, the values are reported as comparable to other lignocellulosic cementitious compounds.

Boix et al. [59] investigated particle pre-treatment systems consisting of alkaline leaching and silanization protocols and the influence on the concrete specimen. The compression strength of concrete, the sugar amount released from the biomass during concrete exposure and the effect on the setting of concrete were determined. *Miscanthus* particles were leached with NaOH and treated with tetraethyl otrthosilicate (TEOS) emulsions at different pH levels (pH4, pH 6, and pH 10). The compression strength of the concrete increased from 2.2 N mm^{-2} to 11 N mm^{-2} for particle treatment by alkaline leaching, followed by acidic silanization. A negative correlation between the compression strength and the amount of sugars released into the concrete water was observed, such that the strengthening effect was attributed to the initial basic sugar leaching and silane coating against degradation in concrete. Fourier transform infrared spectroscopy (FTIR) confirmed the lowest C-S-H inhibition and conductimetry displayed that the setting time for the leached and silanized biomass faster than with pure water. The influence of the treated biomass on the setting of concrete is thus highly complex.

Chen et al. [56] varied *Miscanthus* loading rates and pre-treatment methods for acoustic absorption properties in concrete with a w/c ratio of 0.45. The *Miscanthus* was loaded in volumetric dosages of 10%, 20%, or 30% as different milled fractions and the pre-treatment methods performed included soaking of the fibers and impregnation by cement slurry. The cement mixture was composed of Portland cement and ground granulated blast furnace slag aiming to reduce the alkaline degradation of the biomass. The compressive strength decreased for increasing fiber loading from 13.4 to 2.26 N mm^{-2} for pre-wetted particles of 0-2 mm against the reference with 55.4 N mm^{-2}. Larger particles with 2-4 mm and *Miscanthus* powder followed the same trend with respective strength decreases of 14.8-5.21 N mm^{-2} and 17.58-3.59 N mm^{-2}. Cement impregnated particles decreased in strength with increasing fiber loading from 23.14 to 10.78 N mm^{-2} for 2-4 mm and 23.69 to 14.59 N mm^{-2}. The smaller trend of decay for the cement impregnated fibers, due to reduction of leachates through the encapsulation of biomass. The mechanical properties are nonetheless concluded to be improved compared to other bio-based lightweight concretes.

No general trend is observable for the particle size effects over both pretreatments, except that powder fractions appear to reduce the compressive strength less. The flexural strength of cement impregnated powder samples on the other hand is reported to increased by about 30% compared to pure cement paste. The acoustic absorption coefficients of 2-4 mm prewetted *Miscanthus* fibers displayed an increase in the coefficient for low fiber loading with 10% fiber and a shift to higher frequencies with further increased fiber loading. It was concluded that the open pores and voids introduced by the *Miscanthus* significantly enhance the acoustic absorption properties.

Ezechiels [60] designed *Miscanthus* concrete in a master's thesis. Several aspects of concrete mixing with *Miscanthus* feedstock were addressed, and the main findings are described below. The influence of the water dynamic was researched by comparison between pre-saturated water fibers and excess water to saturate fibers in the concrete mixture. It was concluded that no preference for a specific fiber hydration method exists in terms of concrete compatibility *via* calorimetry. Hence the water demand of fibers can be treated by the addition of a calculated amount of extra water to the concrete mixture.

The influence of different fiber loadings from 2-10% on the concrete was determined [60]. The mortar workability was found to decrease by increasing the fiber loading and compressive and flexural strength were reported to decrease with fiber content. The strength values decreased about 60% when maximum loading of 10% fibers was reached.

Different pre-treatments to reduce the water absorption of *Miscanthus* were tested [60]. Next to physical pre-treatment by fiber densification in a hornification process, encapsulation of particles in a water impermeable or hydrophobic matrix (cement, slag and waterglas, lignin, linseed oil, or for example oxydizing oil) were tested. The most efficient reduction of water absorption was 140% by waterglas pre-treatment and 210% by cement slurry. Due to the incomplete inhibition of the water absorption it may be concluded that no full encapsulation has ensued and pores are still accessible.

4.5.4 Summary and Conclusions: *Miscanthus* Concrete

In cementitious *Miscanthus* materials, variations in the mechanical performance are caused by the complex interaction of the biological feedstock with the chemical setting reaction. The introduction of porous biomass decreases the pressure stability, but increases the acoustic insulation performance of the material. Reinforcing a concrete matrix requires a good particle to matrix bond that does not get disrupted by particle shrinkage or phase separation around the particles. The reported key issue is the water absorption of the biomass that may be countered by particle treatments or by calculated amount of excess water within the cement mixture. Organic leachates from the biomass are identified as detrimental to the mechanical properties. Alkaline leaching treatments to reduce the organic leachates have shown an improvement on the mechanical properties. Treatments to reduce the organic leachates have not shown an improvement on the mechanical properties. The alkaline medium causes biomass degradation into process inhibiting substances; however, the strength of degradation has not yet been quantified. Further investigations should include more effective pre-treatments against water absorption and improved particle to matrix bonds, as well as, systematic studies to quantify the magnitude of the strength reducing effects of the organic leachates.

4.6 Conclusion

This review provides an overview of *Miscanthus* based materials and their conversion from biomass under viewpoints in the scientific community. This review of *Miscanthus* based materials provides an overview of plant specific conversions from biomass to a product in the scientific community with a focus on construction and building materials. The potential feasibility of *Miscanthus* based materials applications has been reported for particleboard and concrete applications, such that persistent CO_2 fixation is possible. While the technical constraints and research needs are currently hampering direct economic applications, key factors for the development of polymer systems, fiberboard production, and concrete have been identified.

The most important limitations regarding the knowledge or technology to convert *Miscanthus* into technically viable materials are insufficient definitions of a usable source material. A targeted pre-treatment to ensure sufficient particle bonding is the key to obtaining a usable feedstock. Further differences in the source material qualities of the different genotypes should be quantified and ranked for their relevance to the applications. The main constraints in knowledge or technology of the conversion of *Miscanthus* to technically viable materials are missing definitions of a viable feedstock. The active and goal oriented *Miscanthus* pre-treatment is the key to obtain a viable homogeneous feedstock for each use. Further differences in the feedstock qualities of the various genotypes should be quantified for the applications. All of the described systems share the bond between the *Miscanthus* particles and the binding system as the main factor for critical functions.

The feasibility of a partial wood replacement for the glued particle boards has been proven. Only MDI glue showed good adhesion, due to the pH-value of the biomass, with the disadvantage of increased costs. The internal bond strength was mainly limited by the parenchyma content; however, can be increased to an acceptable level by increasing the wood content of the boards. Reinforcement of the boards with flax fiber mats led to considerable improvements in MOE and flexural strength. The feasibility of partial wood replacement in adhesive particleboards has been shown. Only MDI glue displayed a good bond due to the pH of the biomass. However, the internal bond strength of the boards was mainly limited by the structural integrity of the *Miscanthus* parenchyma.

The surface area and particle size of hot pressed binderless particleboards have a large influence on the mechanical parameters. A good feedstock can be produced by steam explosion; however, for economic reasons, a substitution of steam exploded raw materials would be desirable. A quantification of the particle parameters obtained by steam explosion may be leading to a substitution by matter of other processes or the valorisation of the residual biomass.

Miscanthus fibers or particles were shown to improve the mechanical performance of polymer systems if compatibility to the matrix was established. Fibers may be compatibilized by maleic anhydride grafts in hydrophobic polymers thereby adding costs during pre-treatment. PHBV may be used without further grafting, however, is not a high volume polymer. The added particles are preferred with a high aspect ratio, whereby the processing into a matrix blend can cause severe fiber degradation, both by heat and by mechanical perturbation. Therefore, analysis techniques for the produced matrix blends need to be developed and qualitative links to the feedstock will need to be established. *Miscanthus* fibers or particles were shown to improve the mechanical performance of polymer systems under certain conditions. The parameters of particle size, mainly in terms of aspect ratios, were identified as key parameters together with the fiber compatibilizer. Additionally, the particle size parameters need to be quantified for feedstock and the produced matrix.

In concrete systems, the water absorption capacity of *Miscanthus* was identified as most easily treated feedstock parameter. The addition of a known amount of excess water was described as the most economic pathway. Further strength reducing effects like organic extractives and alkaline degradation products may be limited by chemical pre-treatments. Alkaline leaching, followed by silanization, was found as a most effective solution to the drawback of processing steps and material costs. However, further strength reducing effects reside in the introduction of structural voids and further analysis of the quantitative performance is required. In concrete systems, the water absorption capacity of *Miscanthus* was identified as relevant feedstock parameter. Different treatments have been attempted in literature. However, there are further strength reducing effects. Structural voids are introduced via the biomass as well as influencing organic extractives and alkaline degradation products. Further analysis of the quantitative performance influence is required.

5. Improving Mechanical Performance of Self-Binding Fiberboards from Untreated Perennial Low-Input Crops by Variation of Particle Size

Outline

Bibliographic Information

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Abstract: Studies on self-binding hot-pressed fiberboards using agricultural byproducts aim to identify alternatives to scarce wood resources. Particle size and mixture significantly impact strength, although direct comparisons are difficult due to differences in study methods. We evaluated fiberboards made from the two perennial biomass crops *Miscanthus* and *Paulownia* and compared them to *Picea* (spruce), using five distinct particle size blends prepared from milled and sieved particles, respectively. The boards were evaluated for their modulus of elasticity, modulus of rupture, reaction to fire, water absorption, and thickness swelling. All specimens exhibited normal ignitability, as defined by Euroclass E according to EN13501-1. The results indicate that mechanical performance improves with increasing density, which correlates with higher proportions of finer particles. Notably, the finer *Miscanthus* blends and all *Paulownia* samples met the modulus of elasticity requirements of EN 622.

5.1 Introduction

The long-term availability of raw wood from forestry in Germany is expected to decrease from $\approx 85 \times 10^6\,m^3a^{-1}$ in 2013 to $\approx 78 \times 10^6\,m^3a^{-1}$ in 2052, with ≈44% of the available wood being spruce (*Picea*) [107]. The regional *Picea* stands have been damaged by recent storms and bark beetle infestations leading to high volumes of calamitous wood and quality loss in the absence of preserving measures [234]. As one of the main cost drivers of particleboard production, next to the used binding agents, is the utilized woody biomass, new alternatives like agricultural residues and self-binding fiberboards have to be considered in view of the increasing resource scarcity and competition to strive for increased sustainability in the construction sector [120, 235]. Perennial biomass crops are currently being investigated as a sustainable and diversified raw material base for various bio-economic applications [12, 236, 237]. Of particular interest are crops with high biomass accumulation on marginal sites, such as the C_4 sweet grass (*Poaceae*) *Miscanthus* with a yield of 10–25 $t\,ha^{-1}\,a^{-1}$ [26], or the deciduous tree *Paulownia* [12] (*Paulowniaceae*) which can be cultivated at 5000 trees ha^{-1} and harvested in a 4–6 year cycles for high biomass production [74]. Due to their rapid growth, both cultures are under investigation for bio-energetic [26,35,74] and bio-refining applications [16,74,76], but material applications for *Miscanthus* [238] and *Paulownia* also play an increasingly important role, for example as basis for particle boards [239]. Both biomass plants could thereby supply biomass for materials applications.

Self-binding fiberboards have been researched to obtain new raw materials for the construction industry [120, 122, 125]. Moreover, it is postulated that the substitution of binding agents could, respectively, reduce the emissions of CO_2 [125, 129] and formaldehyde from wood-based materials [124, 125]. Several agricultural by-products such as oil palm biomass [240], olive tree cuttings [129], wheat straw [241], *Cynara cardunculus* [123], *Vitis vinifera* branches [127], and kenaf core [122,130,131,242,243] have been investigated to find possible applications in self-binding fiberboards in various processes.

The core process to manufacture these new materials is the hot pressing of a lignocellulosic feedstock, where thermomechanical deformation induces the adhesive properties of lignins, either by plastic deformation or reaction [125, 132]. In addition, the particle size of the biomass is important, as sufficient particle contact is crucial for a good binding [120, 125]. To facilitate the binding mechanism, the biomass can be pretreated to soften lignin or release hemicelluloses with different chemical and physicochemical methods [125, 128], like fine milling or grinding [68, 128], extrusion [244], steam explosion processing (e.g., Masonite process) [68–71], or chemical reactions in acid or alkaline media or with oxidizing agents, but at the cost of chemical recovery and environmental impacts [120].

The influence of the density of the self-binding boards on their mechanical properties was already observed in some studies: for steam-exploded *Arundo donax* L., the modulus of elasticity (MOE) could be increased from $\approx 2500\,\mathrm{N\,mm^{-2}}$ [124] at $740\,\mathrm{kg\,m^{-3}}$ to $9552\,\mathrm{N\,mm^{-2}}$ at $1295\ \mathrm{kg\,m^{-3}}$ [245]. Thus, increasing the compaction and density of the boards was found to increase the mechanical strength of the boards [70]. Densities of 810–1306 $\mathrm{kg\,m^{-3}}$ and corresponding MOE values in the range of 1610–6590 $\mathrm{N\,mm^{-2}}$ [69, 71] were observed for boards made from steam-exploded *Miscanthus sinensis*. The steam explosion process generally results in acceptable mechanical strength values, but at the expense of energy and special machinery [122]. Furthermore, investigations were conducted to ascertain the efficacy of incorporating lignin [69] or polymeric methylenediphenyl diisocyanate [124] in order to reduce the quantity of steam-exploded fibers, which are associated with high costs.

Self-binding fiberboards can be produced without any pretreatment methods by only milling and pressing [122, 131]. In the absence of thermophysical pretreatments, the fiberboards display reduced mechanical and moisture stability values due to the hygroscopic properties of the fibers [120, 122]. However, also self-binding boards from finely ground biomass without pretreatment can exhibit higher mechanical strength values when the board densities are increased [240, 246]. Particle size is a primary contributor to mechanical bonding properties, as smaller particles can fill voids and act as adhesive by increasing the total contact area [122, 125, 132, 223, 247].

The main goal of this study is to examine the influence of finer particles on the mechanical properties of the produced fiberboards. Therefore, different blends with increasing mass share of smaller fractions are prepared, which is an approach that has not been studied yet in the literature. Due to the heterogeneous nature of plants, in general, and the complexity of the precise chemical interactions taking place in the bond formation process [128, 248], the comparison of literature values is difficult. This is the first study to directly compare the three different biomasses of calamity wood from *Picea*, and the two perennial biomass crops *Miscanthus* and *Paulownia* in self-binding fiberboards. All three were milled and sieved with common agricultural equipment and processed to self-binding fiberboards without any additional pretreatment. To compare the different products, not only mechanical tests were conducted, but also their reaction to fire and behavior after contact with water (thickness swelling (TS) and water absorption (WA)), since these are important properties for possible future building materials.

5.2 Materials and Methods

5.2.1 Biomasses

The *Picea* (Figure 5.1a) wood was chipped from regional calamity wood with a diameter of 8–23 cm. The *Paulownia* biomass was obtained by chipping new shoots (1-year growth) from a plantation maintained at the Campus Klein-Altendorf (Figure 5.1b). *Miscanthus* × *giganteus* biomass was harvested in April 2022 from a well-established field (Figure 5.1c) at the Campus Klein-Altendorf (University of Bonn, Rheinbach, Germany) with a forage harvester (Krone Big X 480, Krone, Germany) using a cutting length of 30 mm. All biomasses were crushed using a hammer mill (type BHS 100, F.A. Buschhoff, Germany) equipped with a 1.1 mm sieve.

(**a**) (**b**) (**c**)

Figure 5.1: Photographic depiction of of the three different biomass types. (**a**) *Picea* in mixed forest (second level). (**b**) Juvenile *Paulownia* Stand. (**c**) Established *Miscanthus* Stand.

5.2.2 Fractionation and Mixture of Particle Size Blends

Particle fractions for the mixtures were obtained by sieving on a vibrating sieve (ASM 100, S & F). The control of each biomass was determined by utilizing the full throughput of a 0.5 mm square sieve mesh (V0). Five mixtures were prepared from two fractions (0.25–0.5 mm (V1); 0.25 mm (V5)) in varying rations, as shown in the Table 5.1.

Table 5.1: Overview of the preparation of different self-binding fiberboard variants (V1–V5) of *Picea*, *Paulownia* and *Miscanthus*, respectively. The control V0 reflects the native ratio of powder to particles obtained after sieving through a 0.5 mm screen.

Label	**Powder <0.25 mm**	**Particles 0.25–0.5 mm**
V0	Control	Control
V1	0%	100%
V2	25%	75%
V3	50%	50%
V4	75%	25%
V5	100%	0%

5.2.3 Hot-Pressing of Fiberboards

The self-binding fiberboards were produced according to the scheme in Figure 5.2. The powder was hand-formed into a dry mat in a 30 × 30 cm frame and compressed by a plunger in a hot press (Wickert & Söhne, Landau, Germany) at a single step at 150 bar. The core temperature in the mat was monitored with an inserted thermal wire for heat conduction and held at 150 °C for 15 min. The boards were cooled under pressure to 80 °C to prevent explosive vapor release from moisture formed in the boards. Each self-binding fiberboard (Figure 5.3) variant was manufactured in four replicates.

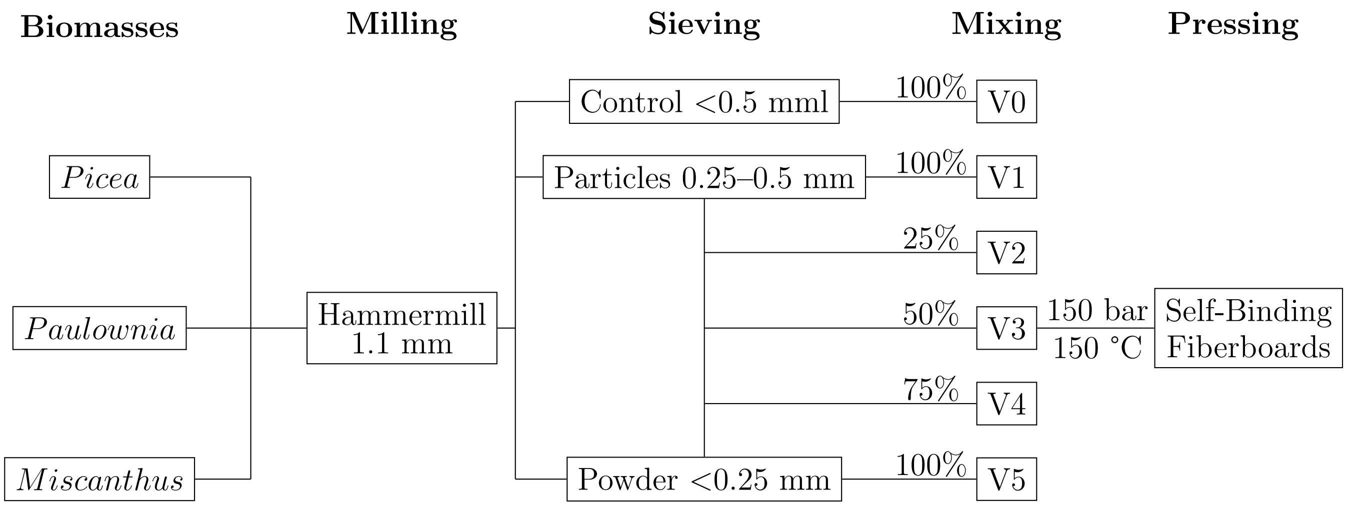
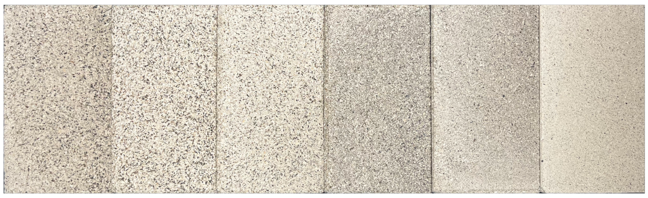


Figure 5.2: Schematic representation of the sequential steps involved in the production of a variant set, with a particular emphasis on the self-binding fiberboard acquisition process. The different particle size variants are composed of: V0 = native distribution < 0.5 mm; and decreasing particle size from V1 = 0.25–0.5 mm (100%) to V5 = < 0.25 mm (100%) in 25% intervals.

(**a**)



(**b**)

(**c**)

Figure 5.3: Self-binding fiberboards of particle sizes V0 (left) to V5 (right) from the three different biomasses (**a**) *Picea*, (**b**) *Paulownia*, and (**c**) *Miscanthus*.

5.2.4 Mechanical Test

The bending strength of the fiberboards was determined using a Hess universal testing machine (HMN10, Hess MBV GmbH, Sonsbeck, Germany) with a 3-point bending test. The tests were performed after conditioning samples of the dimensions 250 × 50 mm at room temperature in approximation of DIN EN 310 [249] and compared to EN 622-2 [250] as requirements for hard boards in load-bearing dry applications (Tpye HB.LA). The raw density of the boards was determined on the mechanical specimen prior to testing. The Modulus of Elasticity (MOE) [N mm^{-2}] was calculated according to Equation (1):

$$\mathrm{MOE} = \frac{l_1^3(F_2 - F_1)}{4bt^3(a_2 - a_1)}, \tag{5.1}$$

where l_1 is the distance between the cylindrical bearings [mm], F_1 is 10% of the maximum force [N], F_2 is 40% of the maximum force [N], b is the width of the specimen [mm], t is the thickness of the specimen [mm], a_1 is the distance the piston travels at F_1 [mm], and a_2 is the distance traveled by the piston at F_2 [mm].

The modulus of rupture (MOR) [N mm^{-2}] can be calculated according to Equation (2)

$$\text{MOR} = \frac{3F_{max}l_1}{2bt^2}, \tag{5.2}$$

where F_{max} is the maximum force required to break the sample [N].

5.2.5 Reaction to Fire

Reaction to fire tests have been performed on 250 × 90 mm samples, which were exposed to a defined propane flame of 20 mm length, held at an angle of 45 °C, for two flame exposure times of 15 s and 30 s in a KBK 917 small combustion chamber (NETZSCH GmbH & Co. Holding KG, Selb, Germany) according to ISO 11925-2 [251]. After the assigned exposure time, the gas flame was removed. The fire tests were evaluated by measuring the height zone damaged by fire where a threshold of 150 mm may not be exceeded for normal ignitability according to the Euroclass system for the classification of construction materials (EN13501-1 [252]).

5.2.6 Thickness Swelling and Water Absorption

Thickness Swelling (TS) and Water Absorption (WA) tests were performed by fully immersing the samples (50 × 50 mm) in 200 mL tap water for 24 h and recording the changes in weight and thickness. The TS was tested after conditioning the samples at room temperature in approximation to the EN 317 [253] test method.

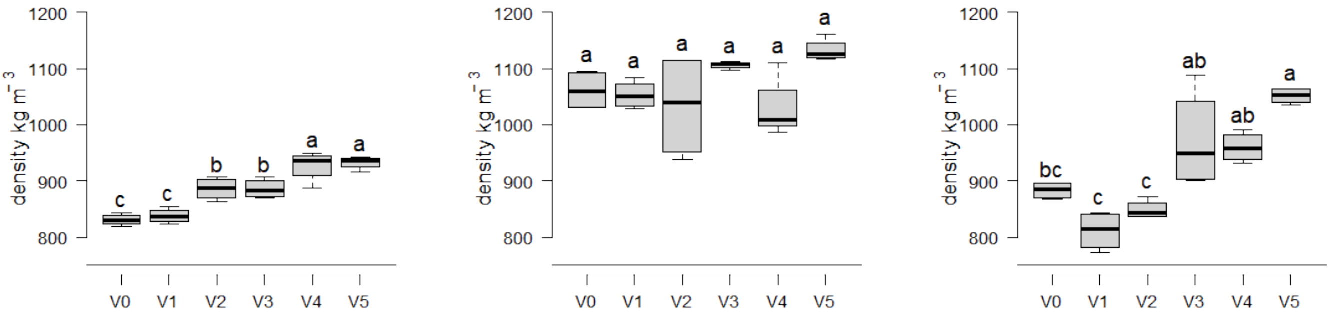
5.2.7 Statistics

Statistical parameters such as mean, standard deviation, one-way analysis of variance (Tukey HSD at $\alpha = 0.05$), linearity (Mandel test for non-linearity at $\alpha = 0.05$) and regression were calculated using the open source programming language R.

5.3 Results and Discussion

5.3.1 Mechanical Evaluation

Figure 5.4 displays an apparent increase in density with increasing powder proportions from V1 to V5 for *Picea* and *Miscanthus*, which is more pronounced in *Miscanthus* from 883 kg m^{-3} to 1052 kg m^{-3} (Figure 5.4c) compared to *Picea* from 831 kg m^{-3} to 933 kg m^{-3} (Figure 5.4a). The three biomasses differ in their density development by increasing the proportion of fine particles. *Picea* and *Miscanthus* display an continuous density increase while *Paulownia* shows no statistically significant increase in density. The detailed numeric values for all variants and their respective performance parameters are noted in Table S1 in the supporting information. *Picea* as wood contains a relatively low proportion of parenchyma cells, whereas *Miscanthus* contains a greater proportion of both cortex and parenchyma cells [154, 213]. A separation of cortex and parenchyma cells by milling and enrichment of the parenchyma in the powder fraction by sieving may explain the larger increase in density observed in the *Miscanthus* powder. It has been reported that softer cell wall structures, such as parenchyma cells, exhibit greater moldability at a given pressure [128], and thus might influence the density as well as the mechanical properties. The highest overall densities are displayed by *Paulownia* at 1062–1133 $kg\,m^{-3}$ in Figure 5.4b illustrating the lack of observed density increase in the tested conditions. This behavior suggests that the softer *Paulownia* [98] particles may be compressed to their limit. This implies that a given density target may be reached with less pressure or particle mixtures containing larger particles when *Paulownia* is compared to *Picea* or *Miscanthus*. The compaction ratio at a given pressure is, therefore, biomass and processing specific.

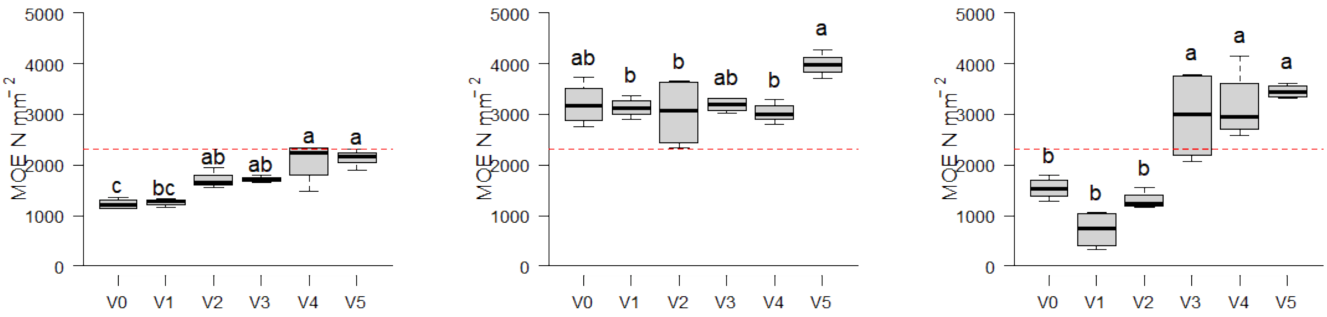


(a) (b) (c)

Figure 5.4: Boxplots showing the density of self-binding fiberboards from the three biomass types *Picea*, *Paulownia* and *Miscanthus* depending on different particle size variants: V0 = native distribution < 0.5 mm; and decreasing particle size from V1 = 0.25–0.5 mm (100%) to V5 = < 0.25 mm (100%) in 25% intervals. Statistical significance is indicated by different letters, representing differences between means based on the Tukey-HSD test at a 95% significance level ($n = 4$).The boxplots consist of the central line representing the median value; the box edges show the 25th percentile (Q1) and 75th percentile (Q3) of the data, with the interquartile range (IQR) as range between Q1 and Q3, representing the middle 50% of the data. The whiskers extend from the edges of the box to the smallest and largest values within 1.5 times the IQR from the quartiles. (**a**) *Picea*. (**b**) *Paulownia*.(**c**) *Miscanthus*.

By varying the particle sizes and biomasses, binder-free fiberboards with significantly different mechanical strengths can be manufactured. An increase in strength is observed with rising powder content for *Picea* (Figure 5.5a) and *Miscanthus* (Figure 5.5c). In general, both exhibit an increase in Modulus of Elasticity (MOE) (Figure 5.5) and density (Figure 5.4) as the mass fraction of particles smaller than 0.25 mm increases. The MOE of the *Picea* biomass demonstrates a notable increase by 68 %, from a lowest value in variant V1 of (1265 ± 73) $\mathrm{N\,mm^{-2}}$ to a highest value of (2137 ± 170) $\mathrm{N\,mm^{-2}}$ in variant V5. The greatest mean MOE increase is observed in *Miscanthus*, by 388% from 717 ± 374 $\mathrm{N\,mm^{-2}}$ in variant V1 to 3455 ± 137 $\mathrm{N\,mm^{-2}}$ in variant V5 (Figure 5.5c). In contrast, *Paulownia* exhibits the highest absolute values with the smallest increase in MOE, as evidenced by the difference between V1 (3124 ± 192 $\mathrm{N\,mm^{-2}}$) and V5 (3886 ± 288 $\mathrm{N\,mm^{-2}}$). This is further supported by the absence of a general increase in board density with increasing dust content, as illustrated in Figure 5.4b.

The resulting diagrams in Figure 5.5 demonstrate that certain fiberboards are suitable according to DIN EN 622-2 [250] for dry, load-bearing applications (type HB.LA) with a required MOE of 2300 $\mathrm{N\,mm^{-2}}$. Although density and MOE display an increase from V1 to V5 in *Picea*, the MOE requirements are not met. In contrast, all *Paulownia* variants readily satisfy the MOE requirements, while the boards made from *Miscanthus* reach the threshold of EN 622-2 [250] with at least an amount of 50 % of fine particles (V3–V5). The relationship between compactibility and density to MOE linear relation should be examined to evaluate additional biomasses for their potential use in self-binding fiberboards. The intrinsic compactibility may serve as an indicator of the potential utilization of diverse biomasses in self-binding fiberboards.

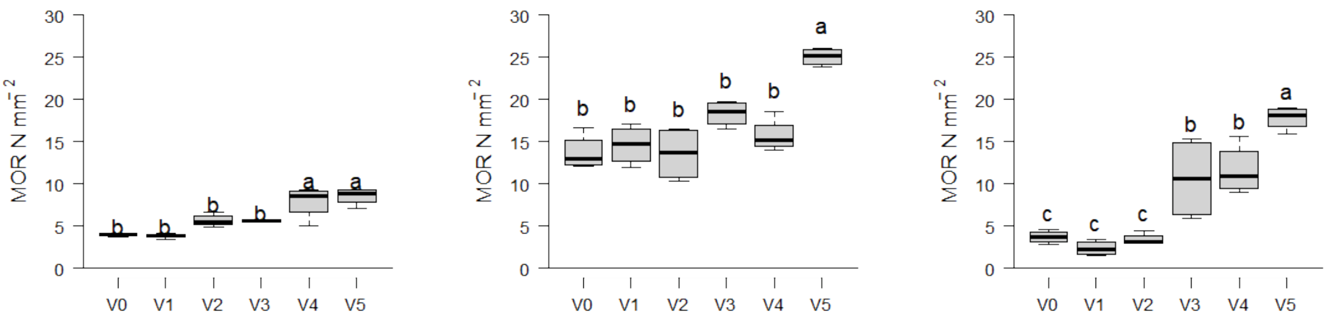


(**a**) (**b**) (**c**)

Figure 5.5: Boxplots showing the Modulus of Elasticity (MOE) of self-binding fiberboards from the three biomass types *Picea*, *Paulownia* and *Miscanthus* depending on different particle size variants: V0 = native distribution < 0.5 mm; and decreasing particle size from V1 = 0.25–0.5 mm (100%) to V5 = <0.25 mm (100%) in 25% intervals. The red line at 2300 N mm^{-2} marks the threshold for dry load-bearing applications according to EN 622 standards. Statistical significance is indicated by different letters, representing differences between means based on the Tukey-HSD test at a 95% significance level ($n = 4$). The boxplots consist of the central line representing the median value; the box edges show the 25th percentile (Q1) and 75th percentile (Q3) of the data, with the IQR as range between Q1 and Q3, representing the middle 50% of the data. The whiskers extend from the edges of the box to the smallest and largest values within 1.5 times the IQR from the quartiles. (**a**) *Picea* (**b**) *Paulownia*. (**c**) *Miscanthus*.

In addition to the observed differences in density, variations in lignin content and structure between *Poaceae*, *Conifers* and deciduous trees [254] could further explain the observed differences in mechanical strength of the boards [128, 132]. Lignin proportions and structures may differ between biomasses [39, 255], particularly in the case of lignin-carbohydrate complexes [256, 257]. For instance, the lignin content is estimated to be between 13 and 27% for *Miscanthus* [237], ≈ 22% for *Picea* [258], and 24 and 26% for *Paulownia* [259]. The prevailing theory of effective bonding posits that a chain of factors must converge to achieve bonding [125]. These include particle size to fill voids, geometry to transfer forces, adhesion by physical forces, and adhesion by complex surface chemical reactions [125]. The latter include plastic deformation, or carbohydrate crosslinking [132]. Further research should include the chemical composition of the biomass used and possibly correlate to the density-to-strength linearity. Especially the biomass harvest time or maturation state may influence the chemical composition and thereby the internal bond strength and thus contribute to feedstock quality.

The Modulus of Rupture (MOR) values increase in a similar manner like the MOE with increasing fineness from V1 to V5, as displayed in Figure 5.6. For *Picea*, the values increased by 130% from (3.8 ± 0.3) N mm^{-2} for variant V1 to a maximum value of (8.5 ± 1.0) N mm^{-2} for variant V5. In the case of *Miscanthus*, the values increased by 640% from (2.4 ± 0.9) N mm^{-2} for variant V1 to (17.8 ± 1.4) N mm^{-2} for variant V5. *Miscanthus* and *Picea* do not demonstrate a statistically significant increase in MOR at low amounts of powder particles (V1–V2). However, at V3, the density of the *Miscanthus* samples (933 kg m^{-3}) is greater than that of *Picea* samples (892 kg m^{-3}) and the mechanical differences become more pronounced. *Paulownia* demonstrated the smallest increase in MOR by only 60% accompanied by the highest means. These values ranged from a minimum of (14.6 ± 2.3) N mm^{-2} in variant V1 to a maximum value of (23.2 ± 4.3) N mm^{-2} in variant V5. As with MOE and density, the *Paulownia* biomass thus forms the fiberboards with the highest mechanical strength. The MOR requirement of 30 N mm^{-2} for type HB.LA in load-bearing applications is not met by any variant, but *Paulownia* V5 meets the threshold of 25 N mm^{-2}, which is required for the general applications outlined in EN 622-2 [250] (type HB). The existing literature indicates that soft biomasses with lower raw densities, such as *Paulownia* at 317 kg m^{-3} [87], can be compacted more effectively than those with higher intrinsic densities such as *Picea* at 370–571 kg m^{-3} [260]. This results in a higher density of the fiberboards produced [239].



(**a**) (**b**) (**c**)

Figure 5.6: Boxplots showing the Modulus of Rupture (MOR) of self-binding fiberboards from the three biomass types *Picea*, *Paulownia* and *Miscanthus* depending on different particle size variants: V0 = native distribution < 0.5 mm; and decreasing particle size from V1 = 0.25–0.5 mm (100%) to V5 = < 0.25 mm (100%) in 25% intervals. Statistical significance is indicated by different letters, representing differences between means based on the Tukey-HSD test at a 95% significance level ($n = 4$). The boxplots consist of the central line representing the median value; the box edges show the 25th percentile (Q1) and 75th percentile (Q3) of the data, with the IQR as range between Q1 and Q3, representing the middle 50% of the data. The whiskers extend from the edges of the box to the smallest and largest values within 1.5 times the IQR from the quartiles. (**a**) *Picea* (**b**) *Paulownia*. (**c**) *Miscanthus*.

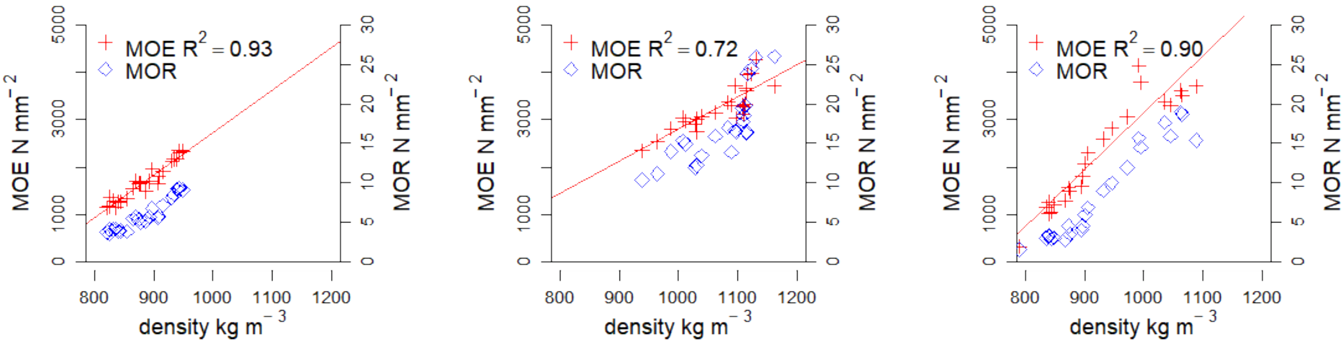
Figure 5.7 and Table 5.2 shows that there are different linear relationships between the material density and its MOE. This conclusion was reached through the Mandel test for linearity. In contrast, the MOR exhibited a better fit with a quadratic model, thereby rejecting the linear assumption. Particle size effects influence the MOR where elongated particles may provide increased numbers of bonding interfaces compared to fines [125]. The increase in the MOR with the amount of fines and the apparent density indicates that the adhesive effect in self-binding fiberboards without pretreatments is mainly dependent on tight particle packing, which in turn indicates a low adhesive strength or insufficient surface coverage in the chosen conditions.

The density values form distinct biomass-specific clusters. The data points for *Picea* (Figure 5.7a) are positioned in the first third of the board density range between 800–950 $\mathrm{kg\,m^{-3}}$. For *Paulownia*, they are positioned in the interval of 950–1150 $\mathrm{kg\,m^{-3}}$ (Figure 5.7b), while *Miscanthus* spans the entire range (Figure 5.7c). The measured data of density and MOE follow a similar pattern as described for binderless boards from mixtures of coarse particles and flour from Kenaf core [122].

The similar behavior of density, MOE and MOR may allow to use the achieved density of the self-binding fiberboard as a quality assessment parameter to project mechanical performance values and set benchmarks required for specific construction applications. With density as a strength determining factor in this production process, further density increases by pressure or the finer milling of particles could increase the mechanical performance without the need for thermophysical pretreatments.

In order to assess the sustainability of self-binding fiberboards produced via this method, it is essential to conduct a life cycle assessment. Accordingly, the biomass feedstock supply process must be documented in accordance with the requisite energy input for comminution and mass streams subsequent to milling and fractionation. Moreover, the overall production process must be balanced.

From the linearity of density to strength properties, it may be possible to establish reasonable limits for a given material and define its applicable use cases such as materials with a focus on high mechanical performance or materials with lower densities for insulation purposes.



(a) (b) (c)

Figure 5.7: Relationship between Modulus of Elasticity (MOE) and density, as well as Modulus of Rupture (MOR) and density for the three biomass types *Picea*, *Paulownia*, and *Miscanthus*. The coefficient of determination (R^2) is provided, when the Mandel test favoured linear over quadratic function. (**a**) *Picea*. (**b**) *Paulownia*. (**c**) *Miscanthus*.

Table 5.2: Tabulated coefficients describing the linear relationship between density and Modulus of Elasticity (MOE) for the three different biomasses *Picea*, *Paulownia* and *Miscanthus* from this study and reported literature values for self-binding *Kenaf* fiberboards. The R^2 indicates the goodness of fit for each linear model.

Biomass	**Slope** [$\mathbf{10^{-6}}$ $\mathbf{N\,m\,kg^{-1}}$]	**Intersect** [$\mathbf{N\,mm^{-2}}$]	$\mathbf{R^2}$	**Source**
Picea	9.29	−6507	0.93	Figure 5.7a
Paulownia	6.82	−4020	0.72	Figure 5.7b
Miscanthus	12.53	−9330	0.90	Figure 5.7c
Kenaf	9.26	−4659	0.96	[122]

5.3.2 Thickness Swelling and Water Absorption

The Thickness Swelling (TS) displayed in Figure 5.8a–c ranges from 218 to 254% for *Picea*, 183 to 292% for *Paulownia*, and 175 to 223% for *Miscanthus*. A reduced TS has previously been linked to increased internal bonding strengths in self-binding Kenaf boards by the application of fine powdered biomass [242]. This study indicates weak internal bonds, as evidenced by the fact that only *Paulownia* shows improvement in TS at V5 (183%) compared to the control V0 (260%) in Figure 5.8b.

To increase the internal bond strength without pretreatments, the adhesive properties may be improved by further softening of the lignin by water addition or increased heat to increase the flowable matrix components as reviewed in literature [125].

The Water Absorption (WA) shown in Figure 5.8d–f ranges from 407 to 518% for *Picea*, 133 to 428% for *Paulownia* and 159 to 446% for *Miscanthus*. *Miscanthus* exhibits a trend of reduced water absorption with increasing amounts of smaller particle sizes. In general the WA is highest for fiberboards made from *Picea*, while boards from *Paulownia* possess the largest TS, which may be related to the generally lower achieved board densities in *Picea*. This is especially true in the case of the highly densified *Paulownia* boards where V0 displays a TS of 260% at a WA of 327% while *Picea* displays a TS of 217% at a WA of 457%. It was also discovered in literature that TS and WA may exhibit different patterns as water accommodation and thus WA may be proportionally increased at lower board densities due to larger voids, while the TS may remain unaffected. This phenomenon is exemplified by Kenaf at densities of 800 $\mathrm{kg\,m^{-3}}$ and TS values of 7–27% and WA values of 45–80% [242] and self-adhesive insulation boards from felted pine bark at 160–300 $\mathrm{kg\,m^{-3}}$ with TS values below 25% with high WA values of up to 380% [261]. Without pretreatments, water uptake by individual particles will cause high-thickness swelling in dense fiberboards. Future research should, therefore, investigate sustainable routes to decrease the TS by reducing the water uptake of the biomass.

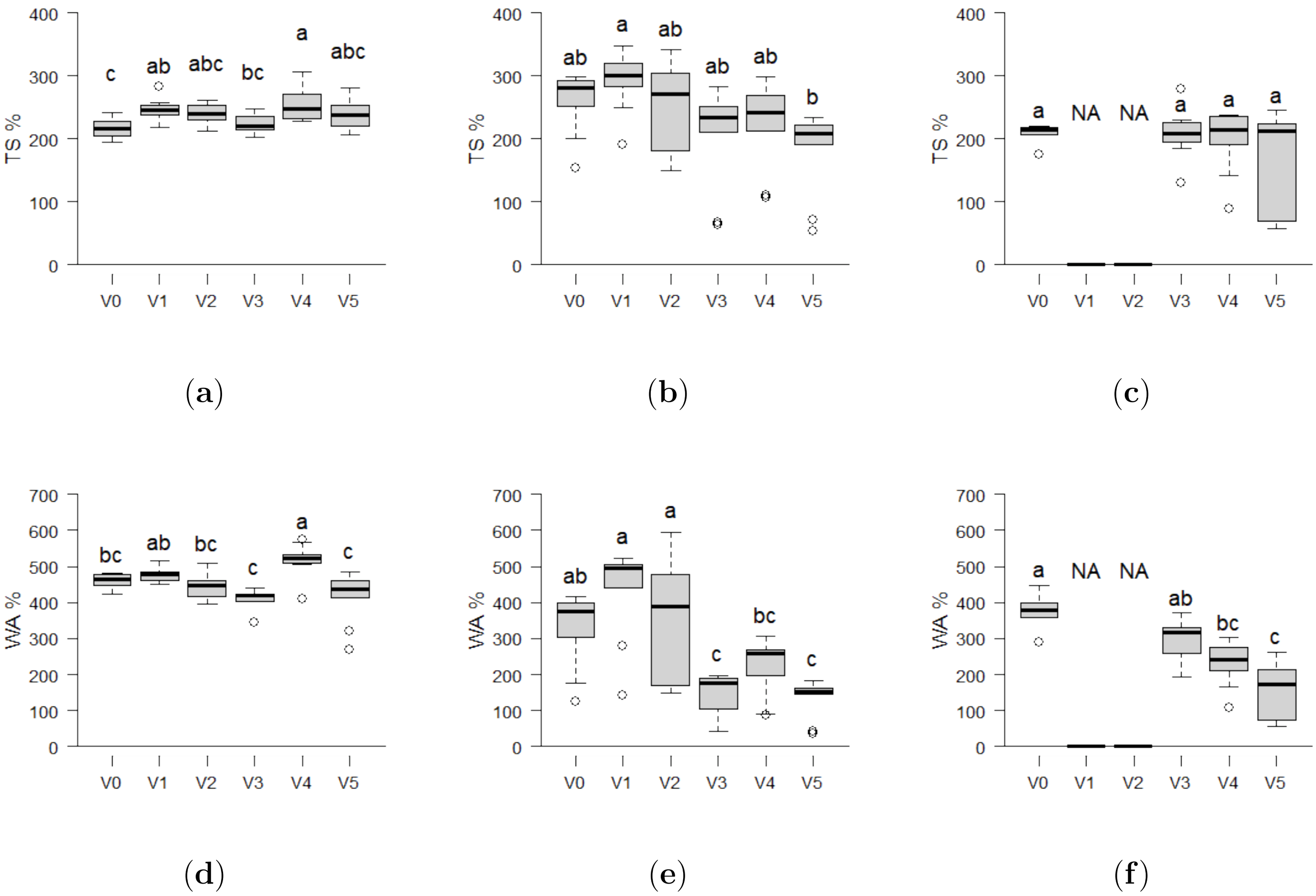
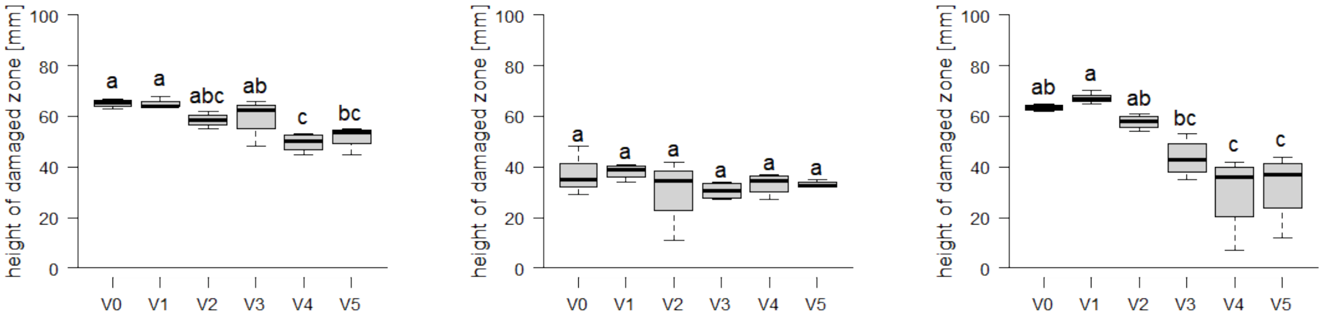


Figure 5.8: Boxplots showing the Thickness Swelling (TS) and Water Absorption (WA) of self-binding fiberboards from the three biomass types *Picea*, *Paulownia* and *Miscanthus* depending on different particle size variants: V0 = native distribution < 0.5 mm; and decreasing particle size from V1 = 0.25–0.5 mm (100%) to V5 = < 0.25 mm (100%) in 25% intervals. Statistical significance is indicated by different letters, representing differences between means based on the Tukey-HSD test at a 95% significance level ($n = 10$). The boxplots consist of the central line representing the median value; the box edges show the 25th percentile (Q1) and 75th percentile (Q3) of the data, with the IQR as range between Q1 and Q3, representing the middle 50% of the data. The whiskers extend from the edges of the box to the smallest and largest values within 1.5 times the IQR from the quartiles. Outliers are marked by an empty circle. Due to the instability of measurements and significant sample losses ($n < 5$), the variants V1 and V2 for *Miscanthus* have been marked by "NA" and been excluded from the plots and subsequent analysis. (**a**) TS of *Picea*. (**b**) TS of *Paulownia*. (**c**) TS of *Miscanthus*. (**d**) WA of *Picea*. (**e**) WA of *Paulownia*. (**f**) WA of *Miscanthus*.

5.3.3 Reaction to Fire

The fiberboards produced from all three biomasses exhibit a measured height of the damaged zone ranging from 10 to 37 mm after 15 s of surface flaming (Illustrated in the SI in Figure S1). For a period of 30 s, the damaged zone range is increased to 30–67 mm, as illustrated in Figure 5.9. This results in a Euroclass E rating according to EN 13501-1 [252], as the 150 mm threshold is not exceeded.

The addition of finer particles resulted in a reduction in the damaged zone heights at 15 s flame exposure for *Miscanthus*, with heights decreasing from 29.9 mm in the control to 13.5 mm in V3 (Figure S1). At a prolonged burning time of 30 s exposure *Miscanthus* a damaged zone reduction from 67 mm (V0) to 32 mm (V5) is displayed in Figure 5.9c. In contrast, the 15 s damaged zone heights for *Paulownia* display the smallest values at 10 mm in V0 (Figure S1), and do not show a statistically significant decrease with increasing powder content at a prolonged flame exposure of 30 s in Figure 5.9b. The smallest damaged zone heights for *Paulownia* fiberboards could be attributed to its high density ≈1000–1100 $kg\,m^{-3}$ and the inherent low ignitability of *Paulownia* due to its special cell structure [262]. The higher flammability of *Picea* could likewise be a attributed to the lower density of the boards (830–930 $kg\,m^{-3}$) or the release of flammable volatiles as thermal degradation products from the coniferous wood [262, 263]. An increase in density and surface closure of self-binding boards has previously been used to explain reduced damaged zone heights [264]. This effect is consistent with the observed density development of all three biomasses, with *Miscanthus* exhibiting a more pronounced effect than *Picea*, while *Paulownia* demonstrates no significant improvement. In tests with lignosulfonate-bound flax plates with a density of 500 $kg\,m^{-3}$, flame exposure of 15 s led to damaged zones of 56 mm, which still satisfied Euroclass E [265]. Lower density presumably leads to more air-filled cavities and increased surface areas, thereby increasing the fuel accessibility.



(**a**) (**b**) (**c**)

Figure 5.9: Boxplots showing the height of the damaged zone at 30 s flame exposure of self-binding fiberboards from the three biomass types *Picea*, *Paulownia* and *Miscanthus* depending on different particle size variants: V0 = native distribution < 0.5 mm; and decreasing particle size from V1 = 0.25 – 0.5 mm (100%) to V5 = < 0.25 mm (100%) in 25% intervals. Statistical significance is indicated by different letters, representing differences between means based on the Tukey-HSD test at a 95% significance level ($n = 4$). The boxplots consist of the central line representing the median value; the box edges show the 25th percentile (Q1) and 75th percentile (Q3) of the data, with the IQR as range between Q1 and Q3, representing the middle 50% of the data. The whiskers extend from the edges of the box to the smallest and largest values within 1.5 times the IQR from the quartiles. (**a**) *Picea*. (**b**) *Paulownia*. (**c**) *Miscanthus*.

5.4 Conclusion

This study proved the suitability of *Miscanthus*, *Paulownia*, and *Picea* as feedstock material for the production of biobased self-binding fiberboards. No special pretreatment of the biomasses is needed; hammer-milling and screening to obtain the desired particle size is sufficient. Specific targets for mechanical performance may be reached by densification. The apparent density can be increased with the addition of fine powder for some biomasses. Future research could include an even finer powder addition, to examine if further improvement is possible. However, finer grinding consumes more energy, so the determination of the mass use efficiency and energy of comminution might be interesting, to assess the sustainability of the proposed method. Comparing the energy requirements for each route of densification is required to assess sustainability. In addition, the variation in the pressing pressure and its influence on the board density and thus, their mechanical properties, is worth studying.

The biomass *Paulownia* produced the strongest boards with the highest densities, while *Miscanthus* exhibited the most pronounced increase in mechanical properties. To understand the role of finer particles in the binderless fiberboards, the composition of the different mass fractions, especially the accumulation of parenchyma, should be investigated. For other biomasses, the specific compactibility and its density increase by powder addition could be used to assess the general suitability for the process.

The self-binding fiberboard behavior exhibited suboptimal performance after contact with water rendering it unsuitable for applications. According to EN 317 [253], the WA and TS values are too high for all boards under consideration. In ongoing research, the potential of hydrophobic treatments is being explored. The MOE demonstrated norm-satisfying behavior in accordance with the EN 622-2 [250] standard for boards in load-bearing dry applications across all *Paulownia* and specific *Miscanthus* mixtures, while all reaction to fire experiments exhibited satisfactory normal flammability according to the Euroclass system for the classification of construction materials. Thus, the self-binding fiberboards produced in this study seem to be a promising approach paving the way for more sustainability in the construction sector.

6. Cup Plant (*Silphium perfoliatum* L.) Biomass as Substitute for Expanded Polystyrene in Bonded Leveling Compounds

Outline

Bibliographic Information

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Abstract: Biomass for non-food applications is considered as a substitute for petro-based materials such as expanded polystyrene (EPS). This research analyzes physical properties of an EPS containing commercial bonded leveling compound (BLC) which was substituted with cup plant (Silphium perfoliatum L.) biomass. Cup plant is a high-yielding biomass plant with several ecological benefits that is yet mainly used for biogas production. Furthermore, the high amount of parenchyma in senescent biomass with its EPS-like structure could be a possible substitute for petrochemical foams in lightweight aggregates. The natural variation in parenchyma content of several European cup plant accessions is promising, regarding the development of cultivars with suitable biomass properties for the proposed material use. Two binders with different proportions of cup plant and EPS were used to produce samples of BLC for thermal conductivity and compression strength tests. The compression strength of 0.92 N mm^{-2} and a thermal conductivity of 84 mW m^{-1} K^{-1} were analyzed and comparable to the commercial BLC. The thermal conductivity within the tested borders appears nearly independent of the biomass content. With increasing cup plant content, the shape characteristics of the lightweight aggregate mix changes towards more elongated aggregates. The mechanical strength and thermal conductivity are highly sensitive to the water demand of the biomass. Direct partial substitution of EPS by cup plant appears feasible and could be a part of the decarbonization of the construction sector.

Keywords: cup plant; *Silphium perfoliatum* L.; perennial biomass plant; EPS; lightweight concrete; insulation; bio-based products; bioeconomy; building and construction materials

6.1 Introduction

Building and construction materials are responsible for 11% of global annual greenhouse gas emissions, and actually building operations are adding a further 28% [119]. Therefore, this industrial sector becomes an integral element of battling climate change. Construction materials, for example, could be used as long-term storage for CO_2 [266]. Atmospheric carbon can be trapped using technical solutions such as carbon capture and storage [267], or through the photosynthesis of plants [268, 269]. Compared to annual plants, perennial crops allow a more sustainable biomass production due to lower nutrient requirements and increased stability against volatile climatic conditions [14, 104, 270–273]. Cup plant (*Silphium perfoliatum* L.) is an undomesticated wild crop [100] with a dry matter yield of 14–25 Mg ha^{-1} y^{-1} [274], if harvested in August for biogas production or of 8.4–14.3 Mg ha^{-1} y^{-1} in case of harvesting in December as a raw material for building materials [104]. This perennial crop can be harvested annually for a period of 15–20 years. Furthermore, cup plant presents several ecological benefits like pollen, nectar and soil health regulation resulting in an increase in biodiversity andminimization of soil erosion [101, 112, 275]. It is also discussed as a high-yielding bioenergy plant for problematic areas such as periodically waterlogged cropland [276]. Furthermore, cup plant has been found to substitute silage maize in biomass yield and quality [277]. Due to these factors, and their high amounts of carbohydrates and proteins, cup plant is currently being used for biogas and fodder [100, 105, 278, 279]. Among other biomasses, cup plant is under investigation as a potential greenhouse gas remedy through the production of biofuels [112]. However, ecological improvement necessitates a biomass feedstock that is procured in a manner that allows an overall increase in sustainability [19, 158, 273, 275]. To take full advantage of the ecological benefits, a late harvest with the least possible agronomical input is necessary [275]. In contrast to biogas applications, late harvests take place after the end of the flowering period so the full pollen and nectar supply is available to pollinators [101]. Furthermore, non-wooden perennial biomass has already been examined as a replacement for woody biomass in the paper industry [20] and particleboard manufacturing [116, 117]. Plant-based materials could play another key role in CO_2 fixation and sustainable construction [280,281]. Due to its positive environmental impact, biomass is suggested as a possible substitute for traditional insulation [280,282,283]. Schulte et al. shows that the perennial grass *Miscanthus* may compete with expanded polystyrene (EPS) for insulation applications [7]. Other applications include light concretes or foam concrete systems. Those are investigated to improve the building materials CO_2 and ecological balance by substituting scarce sand, reducing the compound weight, and improving thermal insulation properties [284]. Integration of natural aggregates in lightweight concrete [154, 285], concrete [2, 155] and foamed concrete has been studied as reinforcements [286]. A partial substitution may be a step towards the development of implemented biomass concretes. The physical performance of this building material substituted with cup plant may suggest different selection criteria for specific applications. Due to visual similarities of the cup plant parenchyma and EPS we assume similar insulating performance for building materials. So, the goal of the study was to investigate if cup plant can be used as lightweight aggregate and partially substitute EPS in bonded leveling compound.

6.2 Materials and Methods

6.2.1 Study Design

The parenchyma contents of several European cup plant accessions were analyzed as quality traits for insulation purposes. Bonded leveling compound served as test application. Its insulation properties and moderate strengths requirements are ideal to analyze the effect of the biomass substitution. The lightweight aggregate constitutes the high-volume compound of the BLC to reduce the overall weight. It was examined if EPS can be substituted by cup plant aggregates and if adverse biomass effects can be reduced by changing the binder. We increased the biomass (0–45 vol%) and thus decreased the EPS share along with the use of two different mineral binders. The thermal conductivity was determined by guarded hot plate analysis and the strength was determined at 20% sample compression.

6.2.2 Plant material

The cup plant biomass was grown at the Field Lab Campus Klein-Altendorf (Rheinbach, Germany). The cup plant biomass used for the BLC samples was obtained from a plot resembling the commercially available feedstock. The field trial was established by planting of plantlets in 2014 (N.L. Chrestensen Erfurter Samen und Pflanzenanzucht GmbH, Erfurt, Germany). The plant material for the comparison of different European accessions derived from the Thüringer Landesamt für Landwirtschaft und Ländlichen Raum (TLLLR). The plants were established at the Campus Klein-Altendorf in 2016. The accessions 'USA', 'Germany', 'Russia', 'Northern Europe' and 'Ukraine', described by Wever et al. [104], were used for the parenchyma quality trait. The annual mean temperature was 9.4 °C with a mean precipitation of 603 mm. The growing season included 165–170 days.

6.2.3 Biomass Preparation for the Construction Material Trial:

The harvest of the cup plant biomass for the BLC samples was carried out in December 2016 (Champion 1200, Maschinenfabrik Kemper GmbH & Co.KG, Breul, Germany,). At this time the relocation of nutrients from the stems into the rhizomes was completed and the plants were senescent. The harvested biomass had a water content of 46% which was reduced to 15% after drying on a drying trailer. To produce a vegetal lightweight aggregate with a similar size distribution comparable EPS the biomass was ground with a hammer mill (BHS 100, Th. Buschhoff GmbH & Co., Ahlen, Germany) equipped with a 10 mm grinding screen. The sieve fraction used for the BLC was 1–6 mm, produced on an oscillating screen (ASM 100, S&F GmbH, Grünkraut, Germany). After sieving no further biomass processing was performed (Figure 6.1).

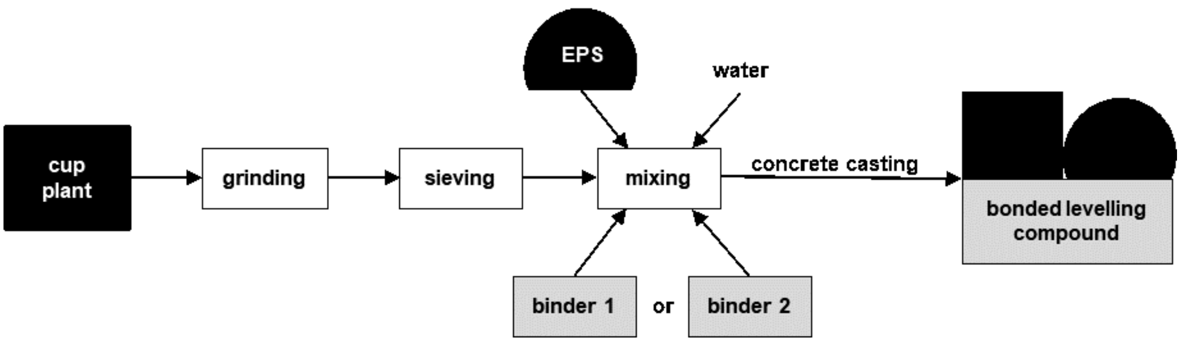


Figure 6.1: Schematic depiction of the study design. Grinding and sieving of the cup plant biomass, followed by mixing with expanded polystyrene (EPS), water, and one of the binders.

6.2.4 Parenchyma Analysis

The biomass used for the determination of the parenchyma content was harvested after senescence in November 2021. The stems were cut 10 cm above ground at the cut height of a field chopper. The inflorescence and remaining leaves were removed. The water content of the stems was 49%. The stems were dried for 24 h at 105 °C to mass stability. Three internodes at different stem heights were used as segments for the parenchyma determination. The low segment was defined as the lowest intact internode above the cut, the high segment was the highest internode, and the third segment was the respective middle internode. From each internode a central segment of 5 cm length was sawed out and the width of each specific stem segment was recorded.

The mass ratio of cortex to parenchyma was established by longitudinal sectioning of the segments and scraping the parenchyma from the cortex pieces. Afterwards both cortex and parenchyma were weighed separately (ME 54TE, Mettler Toledo, Columbus, OH, USA). For the estimation of the parenchyma density the cortex was removed from the lower segments and the size of the parenchyma cuboids was measured. The raw density was estimated by the individual mass of the parenchyma cuboids and their truncated volume (n = 10).

6.2.5 Binder Systems

Binder 1 was extracted from a commercial bonded leveling compound (Fermacell, Bonded Leveling Compound) by sieving. **Binder 2** (Otterbein, PROMPT Fix) was chosen for its high compatibility with biomass aggregates as demonstrated for hempcrete [287]. The binder systems cannot be compared directly, as **Binder 1** is a ready-mix of a cementitious quick setting binder with EPS lightweight aggregates. In contrast, **Binder 2** is a highly hydraulic lime cement powder with high early strength development.

6.2.6 Lightweight Aggregates and Concrete Specimens

The original lightweight aggregates (EPS) of the commercial bonded leveling compound (BLC) were separated by sieving over a 1mm sieve. The mass ratio in the commercial BLC was recorded at 80% binder and 20% EPS. The compositions for the substitution of EPS by cup plant aggregates were measured volumetrically. The biomass ratio ranged between 0–45 vol%, thus the EPS partition shifted between 100–55 vol%. The final list of the lightweight compositions is shown in Table 6.1.

Table 6.1: Volumetric compositions and bulk densities of the lightweight aggregate mixtures containing cup plant (CP) and expanded polystyrene (EPS). 6.1

	Sample Composition [%]				
lightweight component	**CP 0**	**CP 15**	**CP 30**	**CP 45**	**CP 100** *
bulk density [g m^{-3}]	0.045	0.05	0.06	0.07	0.10
EPS [mL]	6500	5525	4550	3575	-
cup plant [mL]	-	975	1950	2925	-

* **CP 100** was used as reference for pure biomass.

All samples were produced using the same weight ratio between binder and aggregate (8:1) according to the commercial BLC. Each sample batch had 500–600 g (6.5 L) lightweight aggregates and 2–2.2 kg mineral binder sufficient material for 4 slabs (15 × 15 × 3 cm) and 6 prisms (4 × 4 × cm). The **Binder 1** samples were produced by mixing 10 wt% lightweight aggregate, containing 0–45% cup plant with 80 wt% mineral **Binder 1**, and water to a w/c of 0.45–0.50. The **Binder 1** sample with 0% cup plant (**CP 0**) was the commercial product and served as control. The **Binder 2** samples were produced likewise, but with **Binder 2** and ascorbic acid (8 g L^{-1}) as a setting delay agent, according to the manual [287], and water, to a w/c of 0.76. To measure the direct substitution potential of cup plant aggregates, the mix-water was given as determined by the product manuals, and no additional water was given to treat the water uptake of the biomass. The lightweight aggregates were mixed and then wetted with 75% of the water. Subsequently, the binder and the residual water were added and mixed for 5 min. The molds for the sample slabs and prisms were filled and compacted by manual agitation. The samples for the compression strength were produced in triplet prism molds, cured at room temperature for 7 days, and finally sawn into cubes (4 cm edge) for compression testing. The compression tests were carried out on a Hess TMN 10 (Richard Hess MBV GmbH, Sonsbeck, Germany) with compression plates at 10 mm s^{-1}. For those tests, a preload of 5 N was used and the force was recorded at 20% sample compression. The slabs for the thermal conductance were cast into slabs, cured for 7 days at room temperature, and sanded flush. The slabs were dried in an oven at 60 °C. During the testing procedure, the slabs were wrapped in cling film to eliminate humidity uptake. The thermal conductance measurements were carried out at an average of 10 °C with 15 K temperature difference using a guarded hot plate apparatus of the type Lambdameter EP500e (Lambda-Meßtechnik GmbH, Dresden, Germany).

6.2.7 Lightweight Aggregate Analysis

Random samples of the lightweight aggregate mixtures **CP 0**–**CP 45** and the raw biomass **CP 100** were homogenized on a sample divider (Retsch, PT 100, Haan, Germany). The analysis of the aggregate sizes was carried out using dynamic image analysis on a Camsizer P4 (Retsch). The shape parameters included in the analysis are Xarea (radius of an equivalent circle), FeMax (length according to Feret), and FeMin (width according to Feret). The width to length ratio (W/L) and sphericity index (SPHT) were calculated from the shape parameters [288]. The water uptake was performed as cyclic water immersion, while the weight was recorded on the universal testing machine (TMN 10, Richard Hess MBV GmbH, Sonsbeck, Germany). After preliminary experiments, the machine was programmed to hold both extreme positions (immersed, well above water) for 30 s, to traverse the Z-axis at 10 mm s^{-1}, and to repeat the immersion process 20 times. Samples for the scanning electron microscope (SEM) were prepared from extracted cup plant parenchyma and EPS by drying in a desiccator for 24 h. The microscopes used were a Phenom ProX (Phenom, Thermo Fisher Scientific, Waltham, MA, USA) and a VHX-7000 (Keyence, Osaka, Japan). The pore size measurements were performed using ImageJ software (v 1.52. URL: https://imagej.nih.gov/ij/download.html, accessed on 1 May 2021).

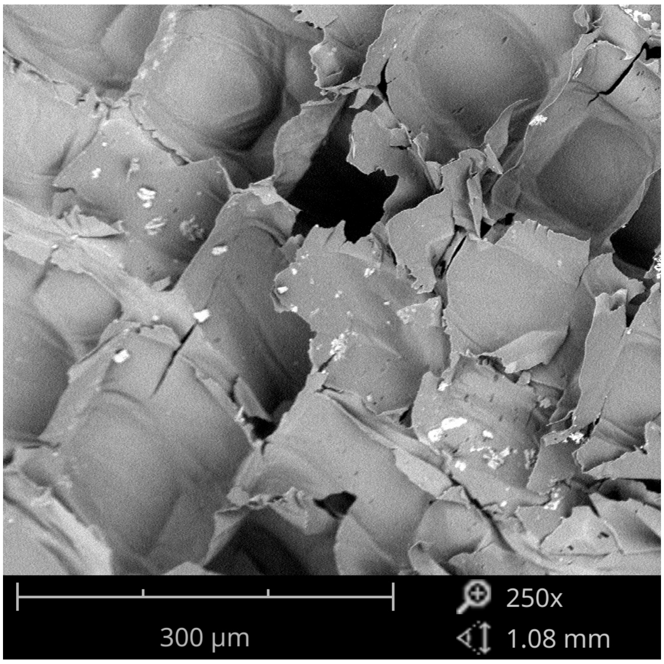
6.2.8 Statistics

Data analysis was conducted in R (R Core Team, 2021) under Version 4.1.0 (18 May 2021). R: A language and environment for statistical computing. R Foundation for Statistical Computing, Vienna, Austria. URL: https://www.R-project.org/, (accessed on 18 May 2021). The used packages were: [289–295]. A one-way ANOVA was performed to compare the effect of biomass ratio on the compression strength and the effect of biomass ratio on the thermal conductivity. The ANOVA was followed by Tukey HSD for homogeneous groupings. The calculations were performed on 6 repetitions for the compression strength values and on 4 repetitions for the thermal conductivity while normal distribution was assumed. The correlation coefficient of thermal conductivity vs. density was determined by the Pearson method.

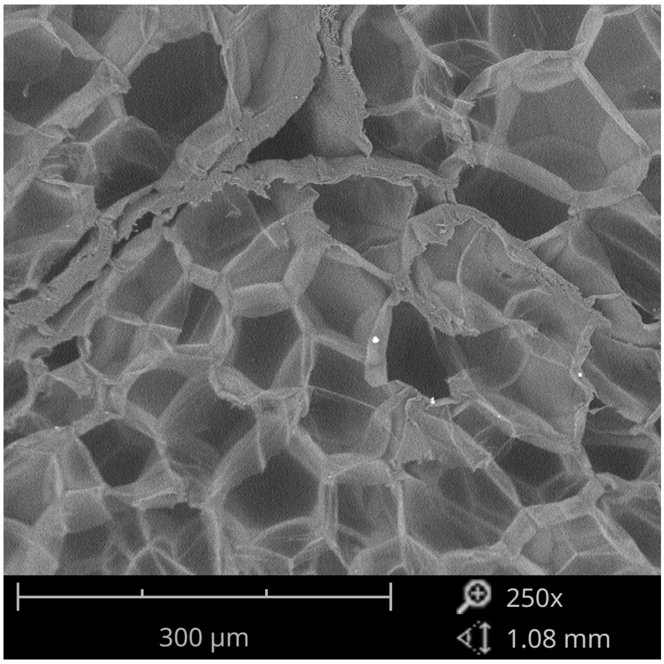
6.3 Results an Discussion

6.3.1 Cup Plant Parenchyma

Due to its foamlike structure, parenchyma seems to be a suitable feedstock for insulation materials. Scanning electron microscopy (SEM) of EPS and cup plant parenchyma cells showed that the macropore diameters range the same order of magnitude for both materials as can be seen in Figure 6.2 . The cell size of the cup plant material (Figure 6.2 a) varies between 141–217 μm and the shape is rectangular. In comparison, the EPS reveals rounded cells with size variations between 44–140 μm in Figure 2b. Neroth et al. assume that pores have to be closed and as small as possible for low thermal conductivity [47]. Therefore, the pore size similarity of EPS and cup plant parenchyma suggest similar thermal conductivities.



(**a**)



(**b**)

Figure 6.2: Structural similarities of (a) cup plant parenchyma, (b) EPS granules under scanning electron microscopy with 250× magnification [10 kV, 60 Pa, image scalebar = 300 μm].

The cross section of the cup plant stem shows the outer cortex as pink tissue after reaction with Wiesner stain and the inner non lignified parenchyma tissue (Figure 6.3). Cross-sections of the stem display a parenchyma area of approximately 44%. Cup plant shows a high volumetric amount of parenchyma throughout the plant stem. The estimated density of parenchyma at 0.041 mg mm^{-3} is in the order of magnitude of EPS with 0.01–0.03 mg mm^{-3} .

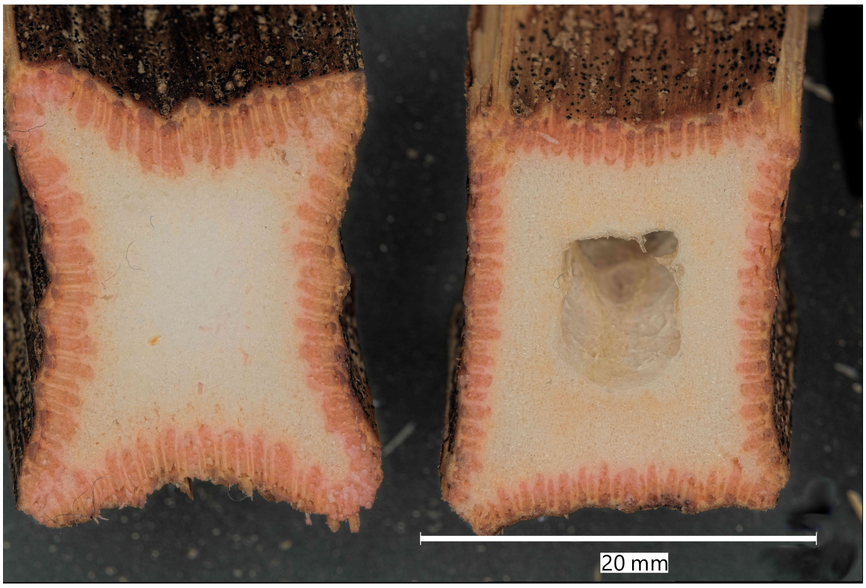


Figure 6.3: Cross-sections of commercial cup plant stems with visible parenchyma and cortex, Wiesner staining of the lignified cortex, under 20× magnification [image scalebar= 20 mm]

As all cup plants, the shoots, from the N.L. Chrestensen plots display a decreasing stem diameter at increasing stem heights. The stem diameter decreases up to 53% from the bottom to top. The absolute gravimetric amount of parenchyma decreases to 38% as shown in Table 2. Due to the low parenchyma density an approximately tenfold amount of cortex is observed. The high standard deviation of stem width and mass is likely caused by the phenotypic variation of the N.L. Chrestensen genotypes. Due to the fact, that the commercially available seed materials (N.L. Chrestensen) is a mixture of several European accessions.

Table 6.2: Biomass qualities of commercially available cup plant material (N.L. Chrestensen) at three different stem heights [n = 32] 6.2

	Diameter [mm]	**Weight Cortex [g]**	**Weight Parenchyma [g]**	**Cortex to Parenchyma**
high	6.8 ± 2.8	0.60 ± 0.34	0.05 ± 0.03	12
mid	10.1 ± 3.3	0.93 ± 0.49	0.09 ± 0.06	10.3
low	12.8 ± 2.8	1.63 ± 0.87	0.12 ± 0.08	13.6

The European cup plant accessions were analyzed for biomass quality in terms of parenchyma quantity. Of the five cup plant accessions displayed in Figure 6.4a) ‘Russia’ displayed a significantly larger parenchyma weight. With 0.121 g the parenchyma weight of ‘Russia’ is 104% higher than the accession with the lowest parenchyma weight ‘Northern Europe’ (0.059 g). This agrees with the previous findings where ‘Russia’ showed the highest stem thickness [104]. However, the highest annual dry matter yield is reported for 'Northern Europe' [104]. Due to the high phenotypic variation of 104% for parenchyma yield, this trait offers the possibility to develop adapted cultivars focusing on a material use of the cup plant biomass. The parenchyma weight and stem diameter show a correlation coefficient of 0.777 over all height levels and all accessions. Therefore, cultivating cup plant accessions with increased shoot diameters define a new cup plant ideal type for material use. The variance of biomass quality in the European gene pool shows that various accessions could be differently suited as feedstock for materials production.

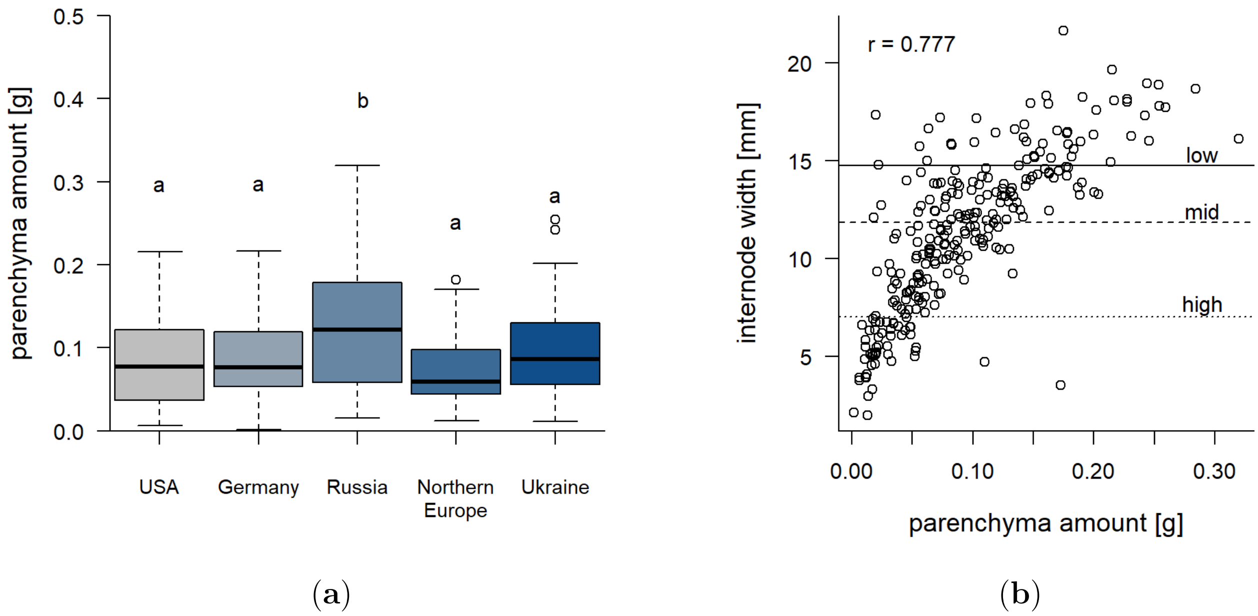


Figure 6.4: Qualities of different European cup plant accessions. The data presented were acquired by surveying internode segments of 5 cm length. Three internodes in different heights (high, mid, low) were examined per stem. In total five accession ('USA', 'Germany', 'Russia', 'Northern Europe', 'Ukraine') with 18 stems each were analyzed. (a) Boxplots ($n = 54$) of parenchyma amounts as a function of their accession. The letters above represent homogenous groupings, calculated via ANOVA with following post hoc test (Tukey-HSD). Different letters indicate significance at $p < 0.05$. (b) Scatterplot of internode width against parenchyma amount ($n = 270$). The correlation coefficient of both parameters equals 0.777 over all five accessions. Horizontal lines display the arithmetic means of internode widths on different positions on the shoot (independent of accession).

6.3.2 Aggregate Analysis

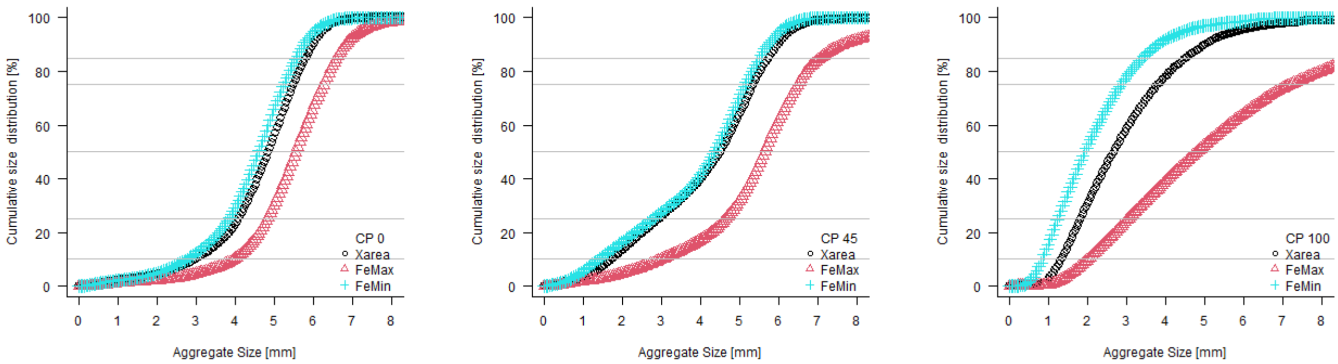
To substitute cup plant in the BLC the shape parameters need to match the original EPS aggregates. The observed cup plant aggregates exist mainly as rod-like shapes of different aspect ratios. Elongated aggregates with low aspect ratios appear to be dominated by the outer section of the cortex. In contrast, the shorter aggregates contain more parenchyma. The resulting biomass is a wide spread of rod-like larger aggregates and more granulated smaller aggregates. In comparison, the EPS used in this study consists of mainly spherical aggregates.

Figure 6.5a shows the high volumetric parenchyma content in the native stem. Shredding of the plant during harvest and processing in the hammer mill causes losses, which can be observed in Figure 6.5a. The overall size of the cup plant aggregates (**CP 100**) obtained by milling and sieving comparable to the expanded polystyrene (**CP 0**) separated from the concrete mix. However, the shape of the cup plant aggregates is inhomogeneous with mainly rod-like aggregates (Figure 6.5a) in contrast to the round EPS (Figure 6.5b).

(**a**) (**b**)

Figure 6.5: Lightweight aggregate examples (a) Cup plant stem and milled sieve fraction; (b) Expanded polystyrene aggregates (sieve fraction: 1–6 mm) [Scalebar = 1 cm].

The composition of the lightweight aggregates shows distinct differences in size (Figures 6.5 and 6.6) and shape distributions for EPS (**CP 0**) (Figure 6.5a), the mixture of cup plant aggregates and EPS (**CP 45**) (Figure 6.6b), as well as pure milled cup plant (**CP 0**) (Figure 6.6c). The aggregate size as cumulative share (Q) of the total distribution allows to represent the influence of the amount of different aggregate sizes on the total composition of the mixtures. The shape of the EPS (**CP 0**) curves (Figure 6.6a) is approximately a parallel sigmoidal curve in all measured shape parameters. This indicates relatively uniform aggregate shape and aspect ratios. The distinct sigmoid shape also shows a sharp aggregate size cut-off, where 10% of the aggregates have a width or equivalent radius below 3 mm. Aggregates above 6 mm have nearly no contribution. The same behavior is observable for the aggregate length but shifted to a size interval of 4–7 mm. In the case of **CP 0** (Figure 6.6c), the sigmoid for the length distribution (FeMax) displays a decreasing slope with increasing aggregate size compared to both other parameters. The size range of the pure biomass up to 3 mm width spans 85%, while it reaches 75% at a length up to 7 mm. For the mixtures of biomass and EPS (**CP 45**), the overall shape of all sigmoid curves shows an increased presence of small aggregates (Figure 6.6b). A shift towards smaller aggregate girths (FeMin) raises the number of aggregates below 3 mm to 25%. The length span of the aggregates is slightly increased (FeMax) from 3–8 mm.



(a) (b) (c)

Figure 6.6: Average development of aggregate shape parameters with increasing cup plant ratio (**CP**) as cumulative size distribution Q. (a) **CP** 0: EPS extracted from the commercial mix; (b) **CP 45**: mix composition of 45 vol% milled cup plant aggregates and 55 vol% extracted EPS; (c) **CP 100**: cup plant aggregates obtained by milling and sieving. The parameters are: Feret minimal diameter (FeMin) as width; equivalent circle radius (Xarea); Feret maximal diameter (FeMax) as length [n = 4].

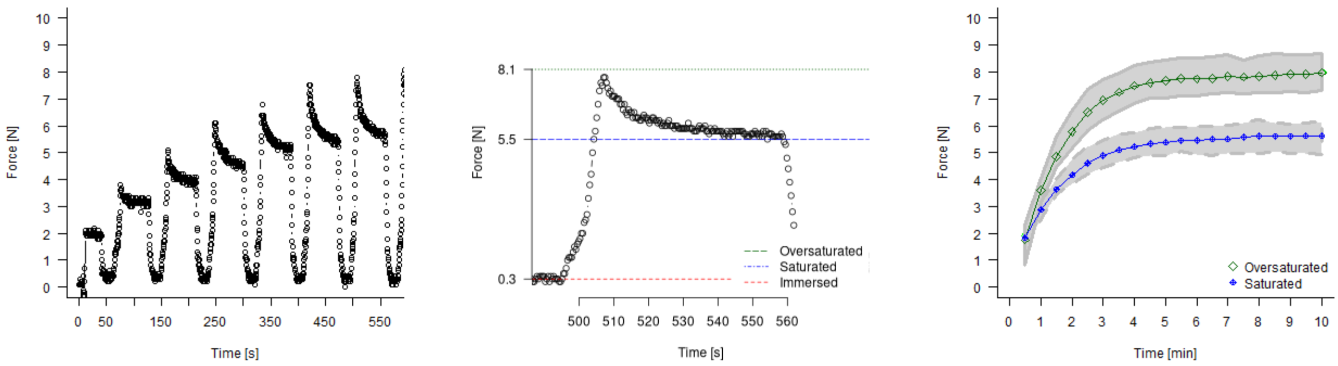
Considering the change in general shape parameters such as the width to length ratio (W/L) and sphericity (SPHT) in Table 3 at the cumulative readout Q, the influence of the biomass in the mixtures **CP 15–CP 45** of up to 85% is minor for the main amounts of the aggregates. The size and shape characteristics are still dominated by the EPS for most aggregates in all lightweight aggregate mixtures used in this study. In order to substitute EPS with an alternative feedstock, this similarity of shapes is necessary to ensure similar workability as rheology and compaction are influenced by the granulometry [296, 297]. Otherwise, a reconstitution of an optimized mixture by predictions methods such as the Andreasen and Andersen model may be necessary [297]. Partial substitution of EPS by cup plant biomass is thus possible, as long as the found feedstock shape parameters are considered.

Table 6.3: Development of aggregate size [mm], aspect ratio (W/L), and sphericity (SPHT, normalized) with increasing cumulative size distribution (Q [%]) of aggregate size (equivalent circle radius) at in-creasing CP levels increasing from **CP 0** (pure EPS) to **CP 100** (pure cup plant) [n = 4].

	CP 0			**CP 15**			**CP 30**			**CP 45**			**CP 100**		
Q[%]	size	W/L	SPHT	size	W/L	SPHT	size	W/L	SPHT	size	W/L	SPHT	size	W/L	SPHT
10	3.0	0.64	0.74	2.2	0.45	0.58	1.9	0.37	0.51	1.7	0.34	0.50	1.3	0.34	0.51
25	4.0	0.76	0.82	3.7	0.64	0.74	3.3	0.54	0.65	2.9	0.45	0.59	1.8	0.34	0.49
50	4.8	0.81	0.86	4.7	0.76	0.82	4.6	0.73	0.79	4.5	0.70	0.70	2.7	0.37	0.48
75	5.5	0.81	0.85	5.4	0.81	0.84	5.4	0.77	0.81	5.3	0.76	0.76	3.8	0.38	0.47
85	5.8	0.83	0.85	5.7	0.80	0.84	5.7	0.80	0.82	5.7	0.75	0.75	4.6	0.38	0.43

6.3.3 Early Onset Water Absorption of Cup Plant Raw Material

The water immersion cycles of cup plant biomass can be seen in Figure 6.7. The individual measurement (Figure 6.7a) consists of 3 cycle stages where 0 N corresponds to the immersed sample, followed by the oversaturated peak, and the wet saturation plateau.



(a) (b) (c)

Figure 6.7: Increasing water uptake as function over time at short wetting intervals. (a) Force against time of cyclic wetting; (b) individual force in dependence of time result of a single soaking cycle. The horizontal lines represent the three stages of soaking in water followed by dripping of adsorbed water until the wet aggregates are saturated with absorbed water; (c) force against time result as mean values of the oversaturated and saturated values. The grey area represents the value range of all samples [n = 6].

The oversaturated peak and wet saturated plateau increase asymptotically and reach a stable value within 5 min of cumulative soaking time as seen in Figure 6.7c. Pude et al. [154] concluded that the most relevant water absorption appears in the first minutes, and pre-soaking of biomass is often performed in practical applications [161, 288, 298]. Water uptake of the biomass has previously been identified as a relevant parameter [154, 160, 165, 285, 299]. However, water absorption is no simple linear process, as physical and chemical absorption processes are contributors as well [298, 300]. The stable water uptake from Figure 6.7c is ≈ 200 wt% of the biomass (300%) for the saturation and ≈ 300 wt% of the biomass (400%) for the oversaturated state. The oversaturated state is of relevance for this application. Water that is superficially adsorbed may disturb the w/c ratio if it is desorbed during the mixing of the concrete. Table 4 with its recalculated w/c values was generated under the assumption that the biomass absorbed sufficient water to reach the saturation plateau during mixing of the wet concrete. Disregarding further water competition between binder and biomass the water availability for concrete during mixing was recalculated. The w/c for Binder 1 starts at a normal level of 0.45 at **CP 0** but decreases rapidly (0.36–0.20) with biomass addition. The same behavior for Binder 2 causes a transition from a high w/c (0.76) to a more usual w/c ratio (0.56) with increasing biomass content.

Table 6.4: Recalculated w/c for each CP level and theoretical water demand of increasing biomass substitu-tion levels from CP 0 (pure EPS) to CP 45 (EPS 55%, cup plant 45%). 6.4

	CP 0	**CP 15**	**CP 30**	**CP 45**
Binder 1 *w/c*	0.45	0.36	0.26	0.20
Binder 2 *w/c*	0.76	0.62	0.59	0.56
Water demand [mL]	-	200	400	600

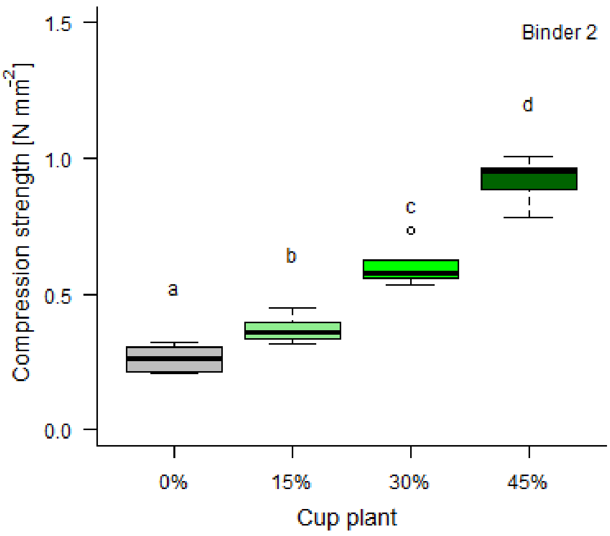
6.3.4 Compression Strength

The compression strength did not display an increase over time in the measured intervals. This is likely caused by the quick-setting nature of both binders and the overall low strength of the BLC. The sample structure consists of the binder covered lightweight aggregates and gas cavities as displayed in Figure 6.8.

(a) (b)

Figure 6.8: 6.8 Samples of bonded leveling compound (a) CP 0 in Binder 1; (b) CP 30 in Binder 2

According to the commercial information [301], Binder 1 is designed to solidify after 6 h and should reach the final compression strength of 0.4–0.5 N mm^{-2} after 28 days. Binder 2 on the other hand is an inherently quick setting binder and is designed to lower a final compression strength above 0.3 N mm^{-2} with biomass, according to the commercial information [287]. The compression strength means of the control (Binder 1, **CP 0**) fluctuated between 0.73 and 0.86 N mm^{-2} at an average of 0.79 N mm^{-2}. With an increase of the cup plant aggregates, the compressive strength of Binder 1 diminished from an average of 0.79 N mm^{-2} (**CP 45**) to 0.40 N mm^{-2} (**CP 15**) and, respectively, further to 0.38 and 0.25 N mm^{-2} for **CP 30** and **CP 0** (Figure 6.9a). The compression strength is in the same order of magnitude as other biomass containing concretes such as *Miscanthus* concretes from Pude et al. [285] (0.28–0.75 N mm^{-2}), waterproofed EPS based lightweight aggregate concretes (0.42–0.47 N mm^{-2}) [302], Hemp lime systems from Benfratello et al. [118] (0.09–0.46 N mm^{-2}) but considerably lower than the higher density (1160–1520 kg m^3) systems with strength values ranging from 2–28 N mm^{-2} reached by Chen [157].



(a) (b)

Figure 6.9: Compression strength values [N mm^{-2}] at 20% compression after 28 days. Samples with 0–45% cup plant mixed with Binder 1 (a) or Binder 2 (b). [The letters a–d represent homogeneous sub-sets of the 28 days strength averages according to Tukey HSD, n = 6]

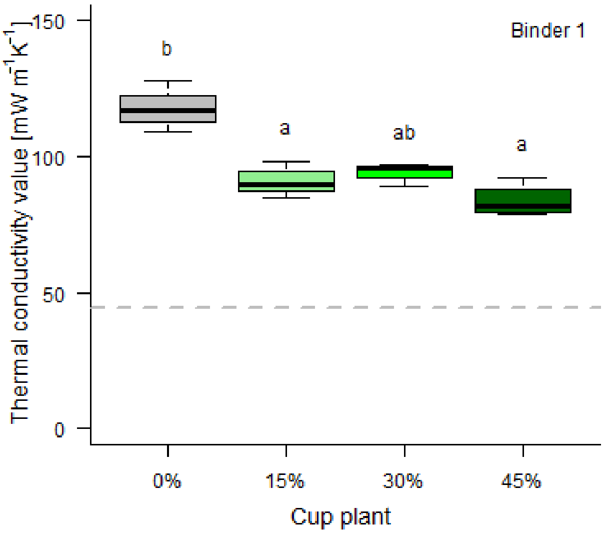
Investigations of the interaction of concrete with biomass extractives have shown that concrete setting will be retarded by different organic species [57, 146, 160–162, 171]. The effective water to binder ratio w/c is also a factor that determines the compressive strength via concrete hydration [57, 154, 297, 299] Even though the binders are setting quickly, the retardation effect should not be dominating since the biomass and the binder systems are still in strong competition for the available water [147]. When compared to Binder 1, the mixture for Binder 2 is especially adapted for biomass aggregates by an increased water amount. For Binder 2 the compressive strength behavior is reversed. The **CP 0** mixture shows the lowest compressive strength at 0.26 N mm^{-2}. With an increased biomass ratio, the compressive strength increases from 0.37 N mm^{-2} to 0.60 N mm^{-2}, and a maximum of 0.92 N mm^{-2} (**CP 45**) (Figure 6.9b). The samples **CP 30** and **CP 45**, therefore, reach the desired strength values above 0.5 N mm^{-2} of the product. The most likely reason for the strength increase is the initial water excess, causing a weakened matrix which is offset by increased water uptake by the biomass. The decrease in w/c is accompanied by a change in workability and mechanical properties as lower w/c ratios correspond to a higher cement stiffness [303]. An increasing water uptake by the biomass may be the main explanation for both the increased compression strength at higher substitution levels in **Binder 2**, as well as the inverted strength behavior between **Binder 1** and **Binder 2**. The **Binder 2** sample is formed with a concrete mix that is made with an initially higher w/c ratio of 0.76 compared to w/c of 0.45 in **Binder 1**. The highest compression strengths of 0.79 N mm^{-2} and 0.92 N mm^{-2} are reached for **CP 0** at w/c of 0.46 in **Binder 1** and **CP45** at w/c at 0.76 in **Binder 2**. Both results correspond to the highest reached density (Table 5) in their respective sets.

Table 6.5: Density values of the compression prim samples for both binder systems at the light aggregates compositions from CP 0 (pure EPS) to CP 45 (EPS 55%, cup plant 45%). 6.5

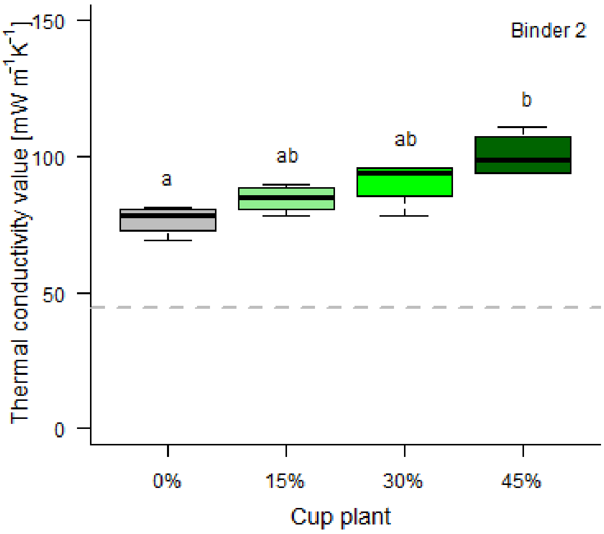
Density [kg m^{-3}]	**CP 0**	**CP 15**	**CP 30**	**CP 45**
Binder 1	604 ± 36	457 ± 39	516 ± 22	466 ± 33
Binder 2	339 ± 37	426 ± 32	564 ± 27	701 ± 47

6.3.5 Thermal Conductivity

The thermal conductivity for **Binder 1** shows two distinct groups with **CP 0** around 117 mW m^{-1} K^{-1} and ≈ 82–95 mW m^{-1} K^{-1} for all other biomass samples, which is shown in Figure 6.10a. A drop in density of the same fashion can be seen in Figure 6.11, as evidenced by the values from 565 ± 36 kg m^{-3} (**CP 0**) to 431 ± 48 kg m^{-3} (**CP 45**). The reduction of the density with biomass addition in **Binder 1** cannot be caused by the inherent density of the lightweight aggregates, as the cup plant biomass displays a higher bulk density than the EPS as it is referenced in Table 1. Hence, the biomass must influence the compaction of the system indirectly.



(a)



(b)

Figure 6.10: Boxplot of the thermal conductivities [mW m^{-1} K^{-1}] of bonded leveling compound slabs produced with cup plant ratios of 0–45%. The grey lines represent reference values of the pure EPS control (45 mW m^{-1} K^{-1}) [69]. (**a**) Thermal conductivity values of **Binder 1** (**b**); Thermal conductivity values of **Binder 2**; differences in values followed by a different letter for each batch are statistically significant at p < 0.05 [(Tukey grouping), n = 4].

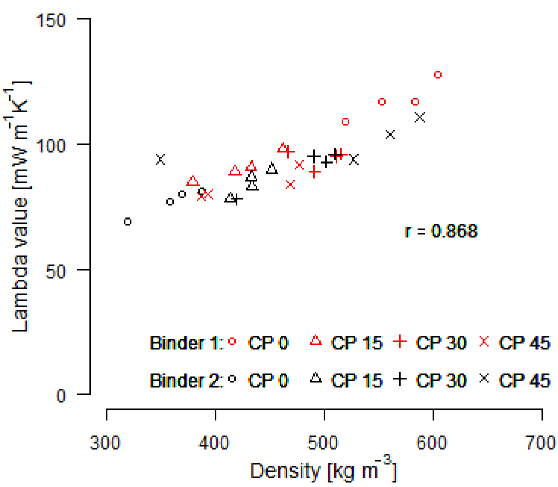


Figure 6.11: Scatterplot of sample density against thermal conductivity values for both Binders at different ratios of cup plant biomass and EPS [n = 4, r = 0.868, $p = 1.245 \times 10^{-10}$].

In **Binder 2**, the density and thermal conductivity both increase with biomass content from 78 to 99 mW m^{-1} K^{-1} and 359 ± 28 kg m^{-3} to 506 ± 107 kg m^{-3} for **CP 0** and **CP 45**, respectively, Figure 6.10b as well as Figure 6.11. The resulting thermal conductivities for both binders are in the same order of magnitude as other insulation concretes found in literature such as hemp lime biocomposite with 83 mW m^{-1} K^{-1} at 231 kg m^{-3} [118], or ultra-lightweight concrete from *Miscanthus* fiber and expanded glass with 90 mW m^{-1} K^{-1} at 554 kg m^{-3} [157]. A strong correlation ($r = 0.86$) between the thermal conductivity and the density can be found in Figure 6.11. These results are in accordance with the generally accepted theoretical framework as insulation materials tend to show lower thermal conductivity with lower density values [304]. In combination with the apparent reversed density effect due to cup plant addition, it must be concluded that compatibility is the main factor determining the thermal conductivity, as is generally the case for insulation concrete [305]However, the span of thermal conductivities for the obtained specimen is sufficient when compared with the commercial reference (120 mW m^{-1} K^{-1}), and follows the same behavior relations as foamed concretes as reported by Samson et al. [305]. The flow behavior and packing density of concretes have been shown to depend on both the water availability and the shape characteristics of the aggregates [296]. Therefore, the difference in water availability by the addition of cup plant biomass impedes the flow behavior and the compaction. Both factors leading to an inhomogeneous density. The thermal conductivity of the composite material is thus a complex function of the lightweight aggregates, the binder system, and the w/c. Future studies of this kind of biomass systems should entail porosity and permeability tests in relation to the parenchyma content of the concrete, as well as a quantification of rheological behavior against bio-aggregate granulometry.

6.4 Conclusions

Cup plant could change the balance of CO_2 emissions for certain construction materials. The local production of the low-density cup plant biomass could be ecologically beneficial compared to the production and transport of petro- or mineral-based lightweight aggregates. The influence of biomass quality traits on the performance of the materials should be further researched. The phenotypic variation of parenchyma content in the European accessions offers the possibility to develop product-adapted cultivars. Long-term breeding goals for materials applications could be derived from the relation of biomass quality and materials performance. Increased stem thickness and the correlated parenchyma increase could be used as selection criteria.

To ensure a sustainable contribution of cup plant use in construction materials the following topics need to be addressed:

- Land use competition between food and biomass production;
- Life cycle analysis of cup plant as industrial raw material;

- Industrial scalability of processing and production of bio-based building materials.

The results indicate that late harvested cup plant biomass could be a biobased substitute for EPS in bonded leveling compounds. The resulting compression strength of 0.92 N mm^{-2} and thermal conductivity of 99 mW m^{-1} K^{-1}, of the sample with 45% cup plant, are within the original product specification. Hence, the biobased samples allow the same applications as the reference product if the binder system is adapted. At the current state, the maximum cup plant content of $\approx$ 45% is limited by the w/c ratio and the rheology of the mix. The short-term biomass water uptake of 200% and the water to concrete (w/c) ratio are the determining factors of the sample density. The density governs both thermal conductivity and compression strength. To obtain a better understanding of the complex insulation system, the porosity of the resulting overall system needs to be determined. The effect of the comminution on the granulometry of the aggregates must be investigated. The resulting changes in rheology must be analyzed so that optimized mixtures can be designed. Experimental studies for the w/c need to be performed to enhance either the thermal conductivity or the compression strength. For practical applications, solid guidelines for the handling of the biomass aggregate and the casting BLC, especially the adjustment of the water to binder ratio, must be implemented. Further studies must address fire behavior, water vapor diffusion, and alternatives to the cement binder, which continues to be the main CO_2 emitter.

Conclusion

7. Discussion and Conclusions

The aim of this thesis was to determine whether perennial biomass crops (PBC) can provide a sustainably produced feedstock suitable for the production of two construction materials, vegetal light concrete and self-binding fiberboard. Biogenic carbon can be temporarily fixed in bio-based construction materials [10], thereby reducing the associated CO_2 emissions compared to construction materials based on fossil carbon sources. Since PBC are predominantly applied in the production of bioenergy, only few examples for the viable application on construction materials are defined.

Therefore a literature review in chapter 3 was carried out to identify the current state of research for the application of *Miscanthus* in different materials applications. The preparation of a suitable *Miscanthus* feedstock in polymer compounds, self-binding fiberboards, and vegetal concrete as two example construction materials, was reported to present technical challenges and knowledge gaps. The chemical and energetic cost of intensive processing and pretreatment methods may limit the sustainability of a biobased construction material in terms of its embedded CO_2 balance.

Based on the literature findings, the guiding principles for the experimental work were set as the exclusion of pretreatments and the production of feedstock by commercially available agricultural machinery. In this thesis, the suitability of a PBC feedstock without extensive pretreatment was verified on self-binding fiberboard and vegetal light concrete. A hammermill and sieve were employed to render different PBCs into a feedstock viable for production of construction materials. The viability of the different feedstocks and of possible mixture adaptations were experimentally evaluated in self-binding fiberboards for the biomasses *Miscanthus*, and *Paulownia* against a *Picea* control and for light insulation concrete as biobased light aggregates obtained from *Silphium*.

Chapter 5 addresses the differences in self-binding fiberboard performance in terms of MOE, MOR, TS, and RTF between the PBCs *Miscanthus*, and *Paulownia* against a *Picea* control that are exerted due to impact of fine particle content on the apparent board density.

Chapter 6 addresses the differences compression strength and thermal conductivity in vegetal light concrete variations by comparing increasing *Silphium* light aggregate contents in two distinct mineral binder formulations.

For each material, different biomass properties were found to influence the structure-property relationships of the resulting distinct construction materials. The performance parameters relevant to each material were determined. This results in a biomass specific suitability for a given process in a given material.

7.1 Application specific Insights and Open Challenges

7.1.1 Self-binding Fiberboard

Self-binding fiberboards constrain a potential production pathway without the need for fossil-based binders, intending in a reduced CO_2 footprint. Improved strength in self-binding fiberboards is obtained at high particle contact area, which are reported to be achieved by fine milling and observed by high densities [125,128]. It has been shown that self-binding fiberboards can achieve standard compliant results or improved performance after fine milling and biomass pretreatments such as steam explosion [68, 306]. To reduce the CO_2 footprint of a produced material, energy intensive processing and special should be avoided. Due to the limiting factors on the processing methods and expected differences between biomasses [128] the following set of questions war raised:

- ***Does commercially available agricultural machinery, such as a hammermill, achieve sufficient particle size reduction to produce viable feedstock for self-binding fiberboards?***
- ***How do Picea, Paulownia, and Miscanthus biomasses differ in their suitability and performance as self-binding fiberboards, particularly when particle size distribution is adjusted?***
- ***How does particle size distribution affect the technical performance of self-binding fiberboards for each biomass type?***

It is technically feasible to obtain a feedstock capable of self bonding from the biomasses *Picea*, *Paulownia*, *Miscanthus* with a hammermill. However, in order to suggest an obtained material for use in construction the final application must be chosen and the required material properties must be met. Fiberboards are generally applied for their mechanical strength. Therefore, the European Norms EN 622-2 for load bearing applications were chosen to provide both some parameters for testing the materials as well as benchmark indicators for performance.

Significant differences in the mechanical performance of the self-binding fiberboards are observed. To determine a limit of applicability, a modulus of elasticity (MOE) MOE of 2300 $\mathrm{N\,mm^{-2}}$ was set as a target for load-bearing applications as specified in the EN622-2 standard. The MOE values ranged from $717-3979\,\mathrm{N\,mm^{-2}}$, while the modulus of rupture (MOR) ranged from $2.4-25.1\,\mathrm{N\,mm^{-2}}$ for all tested variants. It was found that some biomasses, such as *Picea* and *Miscanthus*, do not deliver an inherently viable feedstock that can be converted into a raw material with sufficient mechanical performance. In the first production step, only *Paulownia* displayed MOE values above the threshold.

Assuming that larger particles in the feedstock composition degrade performance by preventing sufficient particle to particle contact, the biomass originally obtained by hammermilling was separated into two distinct size fractions to investigate the influence of particle size distribution per biomass. It was found that the particle size distribution and the biomass used have a significant influence on the strength of materials produced by the dry hot pressing process. The self-binding fiberboards made from different remixed sieve fractions of the three biomasses *Picea*, *Paulownia*, and *Miscanthus* were analyzed for their mechanical performance and apparent density. With increasing fine fractions an increase in apparent density of the self-binding fiberboards is observed. It was observed that the investigated biomasses react with different sensitivities to increasing amounts of fine particles.

Picea does not reach the intended MOE and shows the lowest board densities, while *Miscanthus* reached the acceptable threshold at 50 % of fine particles, with an MOE of about 3000 $\mathrm{N\,mm^{-2}}$ after considerable density increase. In contrast, *Paulownia* readily satisfies the MOE of 3200 $\mathrm{N\,mm^{-2}}$ exhibiting only a slight increase in strength and density due to fine particles after displaying the initially highest densification. For the MOE and MOR properties, a correlation between apparent density and technical performance is observed, where density and MOE are linearly related. By linear correlation of the MOE and apparent fiberboard density, it was observed that each biomass displays a distinct individual linear response. As the apparent density of the fiberboard exerts a significant influence on the MOE and MOR, the biomass compactability is identified as crucial criterion for the suitability of biomasses in self-binding fiberboards.

Next to satisfying the MOE requirements other technical performance parameters of the studied materials remains challenging. The Modulus of Rupture and the thickness swelling which have not reached satisfactory results for the chosen application. The MOR values for MOE-satisfying boards fall within the range of 5 –25 $\mathrm{N\ mm^{-2}}$, which is in line with the literature values for milled systems [120, 130, 242]. The MOR displays a more complex nonlinear relation to the density, but the apparent densities reached up to 1200 $\mathrm{kg\ m^{-3}}$ which is very high in comparison to most other fiberboard materials. The nonlinear relation is presumably due to an increased tensile strength of larger particles in comparison to the adhesion force between fine particles [125]. Future work could entail an enhanced mix design that considers particle shape and optimized packing densities.

Future research should include studies of remaining technical performance parameters, such as, the internal bond as a measure of the particle-to-particle interface, which then could be improved upon. The physical and chemical reactions that give rise to the bonding effect are complex and may depend on Biomass and chemical feedstock composition [128]. The internal bond strength may be influenced by the softening of the lignins, for instance, by addition of moisture, or through reactions between biomass constituents. Determining which effect is the main contributor to the bonding effect is thus highly depended on the production process [125, 128]. Future studies should also seek to derive a correlation between the biomass composition and the mechanical properties, with the aim of defining systematic quality parameters. The biomass composition may thus be quantified in the context of plant senescence or the time of harvest during the year, as well as the storage stability of the harvested biomass.

The thickness swelling (TS) in water is another technical parameter required for the application of fiberboards. The self-binding fiberboards reported in this work display excessive water absorption and thickness swelling, with values ranging from 175 – 292 %. The excessive swelling behavior is frequently observed in untreated self-binding fiberboards [120, 242]. The thickness swelling can be mitigated by reducing the density of the boards, enabling individual particles to swell upon water contact without displacing other particles. This necessitates the formation of a stronger interparticle bond for functional materials due to the reduced particle contact. Enhancing the particle bond strength in the future could, therefore, enhance the MOR and the TS.

Additionally, the TS may be treated through the reduction of the water uptake of the biomass particles themselves. As part of the ongoing research project, a proof of concept for a bio-based and potentially recyclable hydrophobic treatment is being developed [307].

It is feasible to manufacture self-binding boards from PBC through hammermilling. The necessity for sieving into fractions and reconstituting a feedstock with an increased fines content may be contingent upon the biomass and its compactability. Enhancing MOE and MOR may thus be realized via increased apparent density. In the event that future studies determine which parameters of biomass composition have the strongest influence on interparticle binding, quantifiable quality assessment criteria such as the relative proportion of lignin and hemicelluloses can be formulated.

The performance improvement by fine particles suggests that self-binding fiberboards may offer a viable alternative to conventional energetic applications for underutilized biomass byproducts, such as sawdust or prunings. The observed differences in strength between the biomasses were found to be related to their density of the fiberboards, thereby suggesting biomass compactability as potential suitability criterion. To facilitate the future application of these fiberboards as construction materials, it would be beneficial to improve their water resistance by incorporating hydrophobic agents.

7.1.2 Vegetal light concrete

The processing of *Silphium* by hammer mill and sieve can yield particles with the potential to partially substitute for polymeric lightweight aggregates in cement-based leveling compounds, provided that certain adjustments are made. These adjustments include an increase in the water to cement ratio to account for the water absorption of the biomass and the use of fast-setting binders to counteract the reduction in strength caused by organic leaching. This work thus presents an alternative to the application of *Silphium* as a biogas substrate. The strength-reducing effect of organic extractives is difficult to predict, and biomass-cement combinations must be evaluated in a systematic manner. The results of this thesis indicated that a systematic threshold for organic leachates should be established to exclude biomasses from systematic testing in cementitious systems.

Research on vegetal concretes states the two main pathways of interference between biomass and mineral binders. The first is the water uptake of the biomass which can interfere with the setting behaviour of the mineral binder and the rheology by reducing the water availability to the binder. The second is inhibition on the chemical setting of the mineral binders by the release of organic compounds from the biomass. It is established that particularly saccharides and lignin impede the setting of cement and are released by the alkaline degradation of biomass in fresh cement slurry [59, 147, 155, 161]. It is therefore crucial to examine the interaction between a new biomass and freshly mixed concrete experimentally. The research on biobased concrete is frequently concentrated on the utilisation of wood fiber or hemp shives as agricultural residues with the objective of obtaining insulation properties due to the strength reducing influences of the biomass. The reliable introduction of PBC into a cementitious system is a time-consuming and labor-intensive process due to the long setting times of concrete and large number of possible biomass variations involved. The systematic assessment of different biomasses or feedstocks, especially on the accession and minor processing levels, represents a significant challenge for the development and selection of improved procedures. In the existing literature on hempshive-concrete combinations, the biomass compatibility is often adapted through alterations to the concrete, including the addition of setting accelerators and supplementary water. The apparent density of biobased concretes is directly correlated with compressive strength and inversely related to the insulation properties. Consequently, it is thus desirable to identify an acceptable low density and compressive strength for enhanced insulation properties through the reduction of thermal conductivity.

The reduction in density can be achieved through the introduction of porous light aggregates. Some biomasses, such as *Silphium* or Cup plant, contain a substantial amount of pith or parenchyma that resembles the pore structures observed in insulation products like expanded polystyrene, which are commonly utilized as light aggregates. Therefore, it was conjectured that particles produced by comminution of *Silphium* might provide an inherent substitutes for expanded polymer foam granules.

Can Silphium-based aggregates offer a viable, sustainable alternative to polystyrene in bonded leveling compounds?

A commercial polymerfoam-enriched lightweight concrete with reduced strength requirements and defined insulation properties was employed as a model product to assess the viability of *Silphium* as a potential light aggregate. The particle morphology of the biobased light aggregates obtained by hammermilling and sieving were elongated and rod-like in comparison to the round and spherical polymer granules. The light aggregate composition was altered by partial substitution of the polymer foam with *Silphium* particles in ratios ranging from 0 – 45 wt% due to influence of the cup plant particles on the overall particle shape characteristics.

How does the partial substitution of polystyrene balls with cup plant (Silphium) affect the performance and properties of lightweight aggregate mixtures in different binder systems?

The light aggregates were applied under two different binder systems with their respective w/c ratio. By mixing the light aggregates variants with both the original mineral binder without consideration for the added biomass and an biomass adapted binder mixture with increased w/c ratio, it could be shown that increasing the biobased light aggregates had detrimental effects on the performance in the original mineral binder, whereas the compression strength performance of the biomass compatible system was enhanced. In the case of the original mineral binder, the application of any of the *Silphium* substitutions result in a reduction in compression strength to below 0.5 N mm^{-2} which is insufficient to meet the requisite material strength.

The biobased light aggregates with an adapted mineral binder met the commercial non-substituted standard, exhibiting an acceptable compressive strength of 0.92 $\mathrm{N\,mm^{-2}}$ and λ value of $0.084\,\mathrm{W\,m^{-1}\,K^{-1}}$. However, in the adapted binder system, the high amount of water likely resulted in a reduction in density, thereby limiting the combination of compressive strength and low conductivity to high substitution levels of 45 wt% biomass. The values for the adapted binder system with high biomass substitution appear to have improved for both mechanical and thermal conductivity in comparison to literature values at similar density. At comparable density ranges in literature bio-aggregate systems ranging from 0.08 to 0.20 W m^{-1} K^{-1} [118, 166] and compression strengths ranging from 0.5 to 1 N mm^{-2} [118, 166] are reported.

Silphium has the potential to serve as biobased light aggregate for light concrete. The negative effects of biomass water uptake can be mitigated by saturating the biomass with supplementary mix water. The impact of organic leachates can be mitigated through the incorporation of mineral binder mixtures with accelerated setting properties . It is feasible to reach acceptable compression strength and thermal conductivity values for specific applications. It has become evident that the water availability in the fresh concrete mixture serves as the determining factor for the final product density. This is likely to be the result of the mediation of fresh mix rheology and concrete compactibility.

Subsequent studies should employ a systematically coordinated set of experimental conditions. Future research should focus on enhancements through systematic assessments of varying w/c ratios at fixed biomass to binder ratios. A further avenue of investigation would be to examine the setting behavior of mineral binders for individual biomasses, as well as the leaching characteristics of modelled organic biomasses, with a view to elucidating the criteria for the application of these materials in light concrete. While doing so, the rheology of the fresh concrete slurry and the achieved density of the bound insulation material can be determined and correlated with the mix composition.

Furthermore this would permit the definition of a product, thereby enabling the establishment of parameters for permissible feedstock qualities. Consequently, the agronomic influences on biomass quality for application may become evident. In particular, the long-term storability or shelf life of the biomass and its indicators for spoilage, as well as the harvesting settings, methods, and time, may prove to be relevant for the transfer to scaled applications.

Future endeavours could investigate the sustainability of different biomasses if they have to be produced specifically for a product and do not classify as agricultural residue. Especially the consequences in land use change and false incentives should be evaluated.

Prior to formulating recommendations regarding the cultivation of biomasses for use in concrete, it is imperative to conduct a comprehensive life cycle analysis. Furthermore, future life cycle analyses should assess whether the treatment of biomass, the redesign of cement, or the selection of more suitable biomasses would offer substantial environmental benefits.

In circumstances where the utilisation of a rapid-setting binder in conjunction with supplementary saturation water for the biomass negates the inhibitory effects of the biomass itself, an application in insulating light concretes is a viable proposition. Future developments of the product will necessitate the reliable application of the biomass in the selected system. It is recommended that fixed quality assessment criteria be systematically devised based on the water uptake of the biomass, the amount of released hemicelluloses, lignin, and the released organic acids for the application of biomass in concrete. While the complex interactions between the biomass composition and the setting behavior of concrete are tempting to suggest extensive chemical analysis, multidimensional criteria will impact the usefulness in an actual application.

7.2 Generalized Key Insights, and Future Implications

Different biomasses exhibit unique mechanical and functional properties as well as fundamentally different challenges and limitations in each application, as shown for the different materials. The experimental chapters observed how biomass and feedstock formulation impact the performance of the obtained construction materials. Primary processing into specific particle shapes and sizes directly impacts the necessary product formulations to obtain products with acceptable performance. The potential influence of biomass chemical biomass composition on feedstock quality and product performance remains a challenge for individual applications. It is therefore important to understand how the structure to property relations of a construction material are impacted by the characteristics of a given biomass feedstock.

Due to the variability between different PBCs [20,39] and the influence of agronomical procedures on the biomass such as planting density [74], fertilizer application [27,308], and harvest dates [20,27], future endeaveaors have to define permissible feedstock application criteria per application and biomass in systematic experiments.

Materials applications of bio-based construction materials may depend on the evaluation of an application niche where all performance parameters are readily satisfied. While intensified processing or added pre-treatments of the biomass are possible, trade-offs between environmental benefits, mechanical performance, and scalability have to be reckoned with.

Challenges not addressed in this work are the legislative requirements for application of specific materials in construction. Local country building codes as well as user acceptance may differ and hinder the application.

A real application of bio-based construction materials would require a functional value chain consisting of biomass production and value adding steps to achieve a scalable and marketable product. The scalability of production in accordance with availability of resources will be a requisite for development.

The scalability of the described PBCs may be assumed based on the existing and described applications. Production of specific biomasses for material applications raises concerns about land use change and sustainability impacts. Scaling biobased materials production while maintaining environmental benefits is complex. Effects on the biomass cultivation level, such as ecological system services or soil degradation and remediation, may not be easily quantified. Sustainability is thus often reduced to quantify the CO_2 fixation potential of a distinct product in a full life cycle analysis. However, complete sustainability assessments require an analysis of the biomass sources and allocation of by-product, recycling, and waste streams, as well as analysis of societal incentive [10]. Optimization of the production processes and the application of processing sidestreams, such as remaining biomass particles can influence both the ecological as well as the economical assessment of a specific product. A comparison between products requires equivalent functions on the application.

7.3 Conclusions

This work has shown how perennial biomass crops, such as *Paulownia*, *Miscanthus*, and *Silphium*, can be processed by commercially available machinery into a feedstock material for the production of distinct construction materials, such as self-binding fiberboard and bio-based aggregates for light concrete.

Fine particle content and apparent board density as result of biomass compactability were driving factors for the mechanical performance of self-binding fiberboards. The water to cement ratio, the setting speed of the binder and the amount of added biomass has determined the compressive strength and thermal insulation value of vegetal light concrete through its density. In order to successfully develop biobased materials, it is essential to understand how the biomass influences the structure-to-property relations and thus the performance of a material. It is recommended that future research establish quality control parameters for both materials based on particle size distribution and biomass composition.

In order to determine whether products based on biomass offer a substantial sustainability advantage in terms of CO_2, the entire production process, including sourcing, processing, and material performance in application, should be evaluated.

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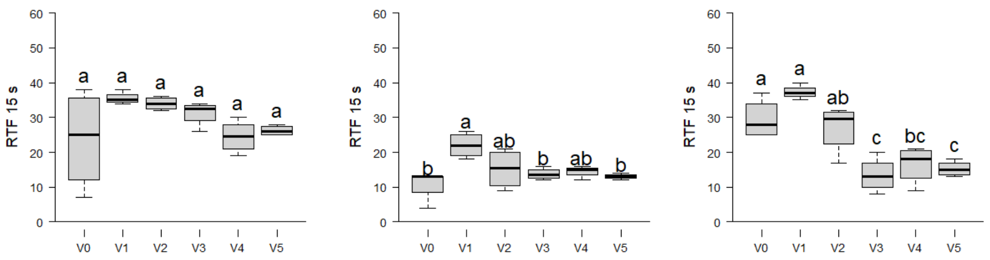
Appendix

A. Supplementary Information: Improving Mechanical Performance of Self-Binding Fiberboards from Untreated Perennial Low-Input Crops by Variation of Particle Size



(a) *Picea* (b) *Paulownia* (c) *Miscanthus*

Figure A.1: **Figure A.1** Boxplots showing the height of the damaged zone at 15 s flame exposure of self-binding fiberboards from (a) *Picea*, (b) *Paulownia*, (c) *Miscanthus*, depending on different particle size variants: V0 = native distribution < 0.5 mm; and decreasing particle size from V1 = 0.25 – 0.5 mm (100%) to V5 = < 0.25mm (100%) in 25 % intervals. Statistical significance is indicated by different letters, representing differences between means based on the Tukey-HSD test at 95 % significance level (n = 4). The boxplots consist of the central line representing the median value; the box edges show the 25th percentile (Q1) and 75th percentile (Q3) of the data, with the IQR as range between Q1 and Q3, representing the middle 50 % of the data. The whiskers extend from the edges of the box to the smallest and largest Values within 1.5 times the IQR from the quartiles.

Table A.1: Numeric means of all measured properties with respective standard deviations of the three biomasses *Picea*, *Paulownia*, and *Miscanthus*

Biomass	**Label**	**density**	**MOE**	**MOR**	**TS**	**WA**	**RTF 15 s**	**RTF 30 s**
		[kg m^{-3}]	[N mm^{-2}]	[N mm^{-2}]	[%]	[%]	[mm]	[mm]
	V0	831.2 ± 10.2	1230.1 ± 104.5	4.0 ± 0.2	217.6 ± 14.7	457.0 ± 20.8	23.8 ± 14.3	65.3 ± 1.7
	V1	837.5 ± 12.8	1265.1 ± 73.3	3.8 ± 0.2	246.3 ± 16.8	476.6 ± 20.7	35.7 ± 2.1	65.3 ± 2.3
	V2	886.3 ± 19.2	1706.2 ± 171.4	5.7 ± 0.8	239.0 ± 16.4	442.9 ± 34.2	34.8 ± 1.8	58.5 ± 2.9
Picea	V3	886.2 ± 17.3	1712.7 ± 65.5	5.6 ± 0.1	223.1 ± 14.3	406.9 ± 35.2	31.3 ± 3.6	59.8 ± 8.0
	V4	927.1 ± 28.2	2070.8 ± 407.1	7.9 ± 1.9	253.7 ± 25.0	518.3 ± 44.4	24.5 ± 4.7	49.5 ± 3.7
	V5	932.8 ± 12.4	2137.5 ± 170.8	8.6 ± 1.0	238.3 ± 22.3	416.3 ± 69.8	26.3 ± 1.5	51.8 ± 4.6
	V0	1061.6 ± 35.8	3197.3 ± 417.3	13.7 ± 2.1	260.1 ± 47.4	327.6 ± 100.7	10.0 ± 5.2	37.3 ± 9.7
	V1	1052.8 ± 25.1	3124.1 ± 192.1	14.6 ± 2.3	292.3 ± 47.9	428.1 ± 131.6	22.0 ± 3.7	38.3 ± 3.1
	V2	1033.1 ± 95.1	3038.5 ± 691.4	13.6 ± 3.2	253.6 ± 67.8	370.5 ± 162.6	15.3 ± 5.7	30.5 ± 13.5
Paulownia	V3	1106.3 ± 6.7	3189.7 ± 142.5	18.4 ± 1.5	204.2 ± 81.4	143.1 ± 63.6	13.8 ± 1.7	30.5 ± 3.5
	V4	1029.1 ± 54.9	3025.7 ± 196.8	15.7 ± 2.0	227.1 ± 68.1	221.0 ± 76.4	14.5 ± 1.7	33.3 ± 4.5
	V5	1132.7 ± 20.2	3979.8 ± 230.4	25.1 ± 1.1	182.4 ± 65.7	133.5 ± 51.1	13.0 ± 0.8	33.0 ± 1.4
	V0	883.0 ± 14.9	1539.7 ± 214.8	3.7 ± 0.8	206.5 ± 18.4	374.6 ± 56.8	29.5 ± 5.7	63.5 ± 1.3
	V1	811.1 ± 35.4	717.7 ± 374.8	2.4 ± 0.9	223.1 ± 10.5	446.2 ± 44.9	37.3 ± 2.5	67.3 ± 2.5
	V2	849.1 ± 16.6	1291.5 ± 182.3	3.4 ± 0.7	207.0 ± 14.1	353.0 ± 45.9	27.0 ± 6.9	57.8 ± 3.0
Miscantus	V3	972.1 ± 88.9	2967.0 ± 906.4	10.7 ± 5.0	206.1 ± 37.6	298.9 ± 59.1	13.5 ± 5.0	43.5 ± 7.5
	V4	960.0 ± 26.8	3155.2 ± 689.3	11.6 ± 2.9	200.0 ± 48.7	232.2 ± 60.0	16.5 ± 5.4	30.3 ± 15.8
	V5	1051.9 ± 14.1	3455.1 ± 137.2	17.8 ± 1.4	175.0 ± 76.5	159.3 ± 70.6	15.3 ± 2.2	32.5 ± 14.2