Master's thesis

Analysis of the ash-softening behaviour of non-woody biomass under the influence of additives

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Kufstein, January 18, 2024

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Abstract

With energy-crisis, climate-change and economic instability being a determining factor of this time, the pursuit of self-sustaining, reliable and renewable energy sources has intensified. Hereby, biomass gasification is a proven technology, both for small and for larger scale plants. The most common type of plant is a fixed bed gasifier with combined heat and power (CHP). At this moment only woody biomass, usually in form of wood chips or pellets, are utilized in this type of gasifier. When looking at the agricultural and forest usage, it becomes evident that there is still is a lot of unused potential, especially for the use of non-woody biomass. This includes both waste materials from existing agriculture, such as straw or corn cobs, and purpose grown crops such as miscanthus and hay. So far, this non-woody biomass has been hampered by the low ash softening and melting points it exhibits. Hereby, ash softens in the gasifier and forms clumps which in turn clog the gasifier. Removal is usually only possible manually, which would lead to plant downtime. A proposed solution to this issue is the use of additives, either in the form of small amounts of non-combustible material, such as kaolin, dolomite or lime, or in the form of blends form non-woody and woody biomass. To assess the usefulness of the different additives and blends, ash softening indices have been developed, they do however still require empirical data regarding their accuracy for biomass gasification. The target temperature of ash softening is >1200 °C.

This thesis aims at providing the empirical data, both regarding the evaluation of the indices and assessing the actual usability of non-woody biomass with additives and in form of blends. Before the experimental part, a background research is conduced. Hereby, an overview over biomass in general, ash composition and behaviour, the pelleting process, the thermochemical conversion, the gasification technology, use of additives, as well as an economic and political assessment is given.

For the experimental part, multiple steps are taken. At first the raw materials, hay, straw, miscanthus, corn cobs and beech wood are cut into serviceable sizes for pelleting. They are then mixed with water and additives, namely kaolin, dolomite and lime, or blended with wood. This mixture is then pelleted. In the next step, the pellets are incinerated following a standardized temperature program, after which only ash remains. In a final experimental step, the ash is analysed using an ash microscope.

The gathered results are as follows: Firstly production of suitable pellets is possible with all tested fuel types and additives. Regarding the ash analysis, the addition of either kaolin, dolomite or lime all lead to a substantial increase in ash softening temperature, above the required threshold. The blending with wood however only yielded one sample with an acceptable softening temperature. The conclusion of this thesis is therefore, that non-combustible additives turn non-woody biomass into a viable alternative to pure woody biomass, woody/non-woody blends however fail to achieve that goal. It has to be noted though, that process conditions in actual reactors differ from those in the ash microscope, thus further research is reacquired.

Kurzfassung

Durch Energiekrise, Klimawandel und wirtschaftliche Instabilität hat sich die Suche nach unabhängigen, zuverlässigen und nachhaltigen Energiequellen intensiviert. Hier stellt Biomassevergasung eine bewährte Technologie sowohl im kleinen als auch im großen Maßstab dar. Üblicherweise werden hierbei Festbettvergaser mit Kraft-Wärme Koppelung verwendet. Diese werden momentan ausschließlich mit Holz betrieben, entweder in Form von Hackschnitzeln oder Holzpellets. Wenn man die aktuelle Nutzung von Wald- und Agrarflächen betrachtet, fällt auf, dass noch eine Vielzahl ungenutzter Potentiale existieren, besonders für die Nutzung halmgutartiger Biomasse. Dies beinhaltet sowohl Abfallprodukte wie Stroh und Maiskolben, als auch gezielt als Produkt gezüchtete Pflanzen wie Miscanthus und Heu. Die Nutzung solcher Halmgüter wird bislang durch die niedrigen Ascheschmelzpunkte eingeschränkt. Dabei erweicht sich die Asche im Vergaser und bildet Anbackungen, welche in Folge den Vergaser verstopfen können. Die Entfernung solcher Anbackungen muss händisch erfolgen, was in weiterer Folge einen Anlagenstillstand bedingt. Eine mögliche Lösung dieses Problems stellt die Zugabe von Additiven dar, entweder in Form von nicht-schmelzenden Materialien wie Kaolin, Dolomit oder Kalk, oder die Herstellung von Mischungen aus Holz und Halmgütern. Zur Abschätzung der Wirksamkeit dieser Additive existieren Indizes, deren Anwendbarkeit bei der Vergasung jedoch noch weiter untersucht werden muss. Die Zieltemperatur der Ascheerweichung ist >1200 °C.

Diese Arbeit hat das Ziel empirische Daten für die Abschätzung der Indizes und für die tatsächliche Verwendbarkeit der additivierten Halmgüter sowie der Holz/Halmgutmischungen zu generieren. Zunächst werden dabei die theoretischen Hintergründe beschrieben. Dabei wird ein Überblick über die generellen Eigenschaften von Biomasse, Aschezusammensetzung und Verhalten, Pelletieren, thermochemische Umwandlung, Vergasertechnologie, die Verwendung von Additiven, sowie eine wirtschaftliche und politische Einschätzung erstellt.

Der experimentelle Teil läuft in mehreren Schritten ab. Zunächst werden die Rohstoffe Heu, Stroh, Maiskolben, Miscanthus und Buchenholz zerkleinert. Anschließend werden Mischungen mit den Additiven Kaolin, Dolomit und Kalk, sowie Holz/Halmgutmischungen hergestellt. Die Mischungen werden anschließend pelletiert. Im nächsten Schritt werden die Pellets nach einem standardisierten Temperaturprogramm verascht. Abschließend wird die Asche im Aschemikroskop analysiert.

Folgender Ergebnisse wurden erfasst: Zunächst zeigt sich, dass die Herstellung von geeigneten

Pellets für alle Mischungen möglich ist. Bei der Ascheanalyse führt die Zugabe von wahlweise Kaolin, Dolomit oder Kalk in allen Fällen zu Ascheerweichungstemperaturen über dem Zielwert. Bei Holz/Halmgutmischungen erreicht allerdings nur eine einzige Probe eine Temperatur über dem Zielwert. Dementsprechend kann die Schlussfolgerung gezogen werden, dass additivierte Halmgüter eine valide Alternative zu Holz darstellen, Holz/Halmgutmischungen verfehlen dieses Ziel jedoch. Es ist außerdem noch anzumerken, dass die Prozessbedingen in tatsächlichen Vergasern von jenen im Aschemikroskop abweichen, daher ist weitere Forschungsarbeit nötig.

Contents

List of symbols and abbreviations

Abbreviations

CHP	Combined heat and power
DIN	Deutsches Institut für Normung e. V.
DT	Deformation temperature
e.	expected
EN	Europäische Norm
FT	Flow temperature
HT	Hemispherical temperature
ISO	International Organization for Standardization
m.	measured
n.a.	not available
n.c.	not considered
OS	Original sample
PPE	Personal protection equipment
SST	Shrinkage starting temperature
ST	Spherical temperature

Physical quantities and units

λ	Excess air number
m_B	Mass of dry biomass in kg
m_W	Water content in kg
u	Moisture content for woody biomass in %
w	Moisture content for non woody biomass in %

%	Percent
F	Form change factor
H	Enthalpy in $kg \cdot m^2 \cdot s^-2$
MW	megawatts
wt%	Mass fraction

Chemical elements and compounds

H	Hydrogen
C	Carbon
N	Nitrogen
O	Oxygen
Mg	Magnesium
Al	Aluminium
Si	Silicon
P	Phosphorus
S	Sulfur
Cl	Chlorine
K	Potassium
Ca	Calcium
Ti	Titanium
V	Vanadium
Mn	Manganese
Fe	Iron
Co	Cobalt
Ni	Nickel
Cu	Copper
Zn	Zinc
As	Arsenic

Mo	Molybdenum
Cd	Cadmium
Hg	Mercury
Pb	Lead
Sb	Antimony
Al_2O_3	Aluminium oxide
$Ca(H_2PO_4)_2$	Monocalcium phosphate
$Ca(NO_3)_2$	Calcium nitrate
$Ca_2(PO_4)_2$	Tricalcium phosphate
$Ca_5(PO_4)_3OH$	Hydroxyapatite
$CaCl_2$	Calcium chloride
$CaHPO_4$	Dicalcium phosphate
CaO	Calcium oxide
Fe_2O_3	Iron(III) oxide; magnetite
K_2CO_3	Potassium carbonate
K_2O	Potassium oxide
$K_2Si_2O_5$	Potassium silicate
K_2SO_4	Potassium sulfate
$KAlSiO_4$	Kalsilite
KNO_3	Potassium Nitrate
MgO	Magnesium oxide
N_2O	Nitrous oxide
Na_2O	Sodium oxide
NOx	Nitric oxide (NO); nitrogen dioxide (NO 2)
P_2O_5	Phosphorus pentoxide
SIO_2	Silicon dioxide
SO_3	Sulfur trioxide

TiO_2	Titanium dioxide
KCl	Potassium chloride

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1. Introduction

1.1. Motivation

Over the past decades, energy availability, prices and sustainability have been hugely discussed topics. From the oil crisis in the 70s, to the most recent energy crisis starting with COVID-19 and later exacerbated by the Russian invasion of Ukraine, the pursuit of cheap, reliable and independently accessible energy sources has be a primary objective of nations and companies alike. In this regard, renewable energy sources have the advantage of often being locally available, without import-dependence on often unstable, authoritarian countries. Furthermore such power plants are frequently small scale, which reduces their susceptibility towards disaster, terror attacks or military strikes. [1] [2]

One example for such decentralized renewable energy sources are combined heat and power (CHP) plants with gasification. At present, those power plants are fueled with wood, which is a proven yet criticized fuel source. The two main points of critique towards wood are the long regrowth time of years to even decades, an the exploitation of protected wood areas through the use of questionable political deals between companies and politics. Because of that, non-woody biomass, such as energy-grasses or agricultural waste products could be a viable alternative to wood. [3] [4] To asses the potential and viability of non-woody biomass for CHP use, TU Wien conducted research on behalf of the the Glock ResearchLab for their sub-company Glock ecoenergy. [5]

In this report, considerable, albeit regionally different potential for an expansion in biomass technologies, was found. When looking at the energetic usage of agricultural and forest area in Austria, large agricultural potential is still untapped in parts of lower and upper Austria, while considerable potential remains unused in Styria and Carinthia. Generally, woody biomass is already well established in use, while non-woody biomass faces a number of issues which are currently preventing the wider use as fuel in biomass gasifiers. One of the major issues are the ash softening and melting, which occur at much lower temperatures compared to woody biomass. This ash softening and melting leads to the formation of clumps inside the gasifier, which can in turn clog the reactor and bring the whole operation to a halt, since this clogging requires manual clearing. Based on the potential it was decided to launch research projects in order to solve the ash melting/fusion issues and ultimately, turn non woody biomass into a viable alternative fuel sources. [5]

One proposed solution to ash melting is the use of additives, which are added during the production of pellets. These additives are inert materials, such as limestone, whose purpose is an increase of ash softening temperature through various chemical and physical effects. An analysis of the theoretical effects of different additives was done by Lessig [6]. Hereby, indices to asses the expected changes of ash softening behavior were calculated. An additional potential solution is the creation of blended pellets from woody- and non woody-biomass, since woody biomass is already established as a viable fuel source. This thesis serves as a continuation in that research effort, with practical experiments to further analyse the viability of non woody biomass, as well as to review the accuracy and viability of the index calculations. To achieve this, practical experiments are conducted, starting with the preparation of the raw material, the production of pellets, all the way to the incineration and subsequent analysis of pellets and ash.

1.2. Aim & scope

Overall this thesis consists of two major parts, a literature research to present the fundamentals, state of the art of technology, and political aspects of biomass pellets, with special emphasis on non-woody biomass, and an experimental part with the production and analysis of such pellets.

The theoretical part consist of seven chapters, starting with the fundamentals of biomass and ash, over the mechanical processing methods and the thermochemical mechanisms happening in the back, towards the gasification technology, the use and effects of additives and finally the political debates and lawmaking regarding the use of woody- and non-woody biomass. For this thesis three research questions are established:

- Can the theoretically investigated enhancements of ash melting temperatures for non-woody biomass fuels mixed with additives be confirmed experimentally?
- Does the estimation of the ash melting behaviour through the calculation of fuel indices provide a sufficient accuracy?
- Could the use of additives in non-woody biomass fuels for CHP application with fixed bed gasification be a technical, ecological and economical alternative to woody biomass?

The experimental part of this thesis focuses on answering those questions. For this some preparations are necessary. Particularly important is hereby the creation of serviceable pellets out of 4 main fuel types, hay, straw, corn cobs and miscanthus, with the addition of the different additives, namely kaolin, dolomite and lime, as well as the creation of pellets blends from the main fuel types and beech wood. Serviceable is hereby defined as mechanically stable enough to not crumble upon transport, as well as the avoidance of any mold or fouling. Such serviceable pellets are the foundation for any further testing.

The whole practical process, from the staring raw material towards the final step, the molten remnants of the sample, is shown in fig.1

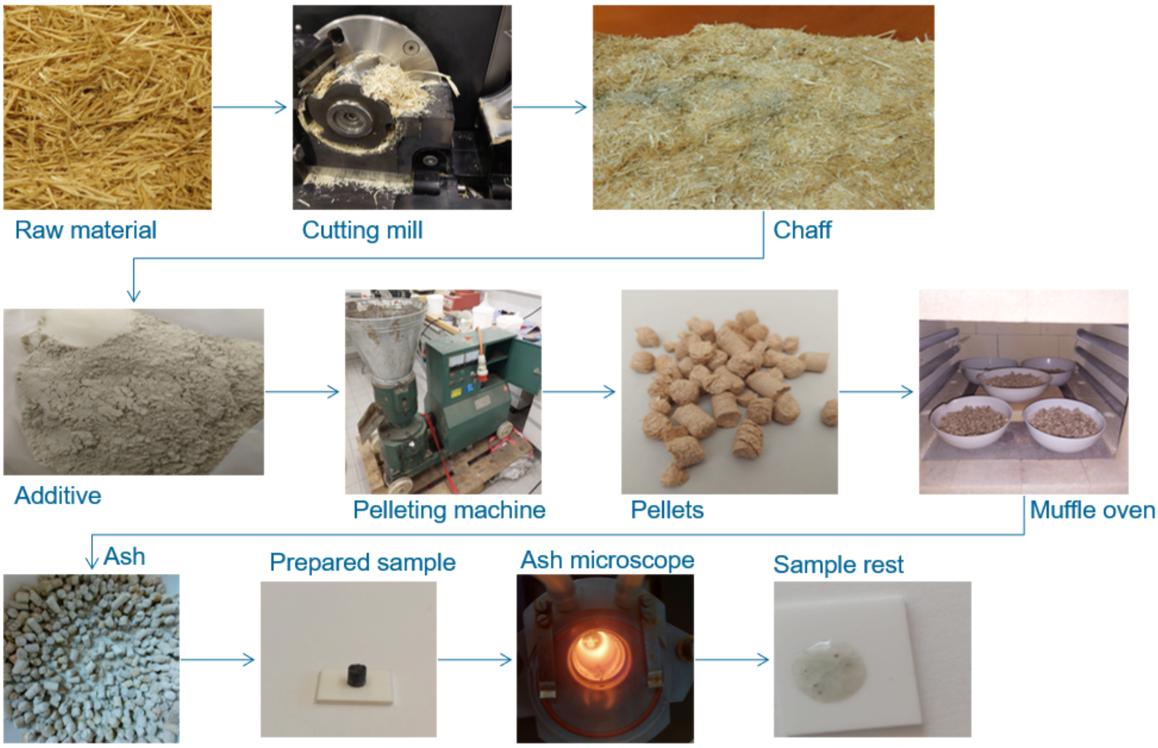


Fig. 1.: *Overview of the pellet production and analysis process*

To actually answer the research questions analysis of the ash softening and melting behaviour is conducted according to the EN ISO DIN 21404 [7] and EN ISO DIN 51730[8] through use of an ash microscope. For this, a total of 20 different fuel compositions are produced and each one is analysed at least 2 times within the ash microscope to create comparative values. Those samples contain varying amounts of additive in order to evaluate the generally necessary quantities. The results are then to be compared both to each other and to the index values, provided by [6], with the target number being ash softening temperatures of greater than 1200 °C, as this would be the requirement for reliable use in biomass gasifiers.

2. State of the Art

2.1. Biomass

Biomass is a loose term which contains numerous materials. In general, those are plants, animals and fungi, their residues and remains, as well as man made materials based on those sources. For further classification biomass is divided into three main groups, primary secondary and tertiary biomass.[9] The definition of biomass is not limited to solid material, but also includes liquids like oils or gases like the gaseous remains of biomass decomposition and also includes both living and dead biomass. Fossilized remains of biomass like coal or petroleum are not included within the definition.[10]

Primary biomass contains all biomass which directly utilises the sun through photosynthesis. Apart from plants themselves, this also includes materials and residues based on plants, for example timber or straw.
Secondary biomass contains biomass from organisms which only indirectly utilize sunlight, which in turn means animals as well as animal residue like manure.
Tertiary biomass contains industrial products based on both primary and secondary biomass, this includes paper and cotton based fabric.[9]

Additionally, biomass may be classified according to its origins into 6 categories, as shown in figure 2:

- Herbaceous biomass contains all types of agricultural products, including grains, oil and nutshells, bagasse, straw, as well as manure from various livestock.
- Woody biomass, also known as forest biomass, contains whole trees, twigs, barks, waste-wood, as well as waste products from the timber-industry such as sawdust or mill scrap.
- Municipal waste biomass contains waste food, sewage products and refuse-derived fuel.
- Contaminated biomass contains sewage originating from industrial installations.
- Aquatic biomass contains all flora and fauna found in aquatic ecosystems, for example algae and fish remains.
- General biological biomass or biomass mixtures contains everything that does not fall into the previously mentioned categories.[10][11]

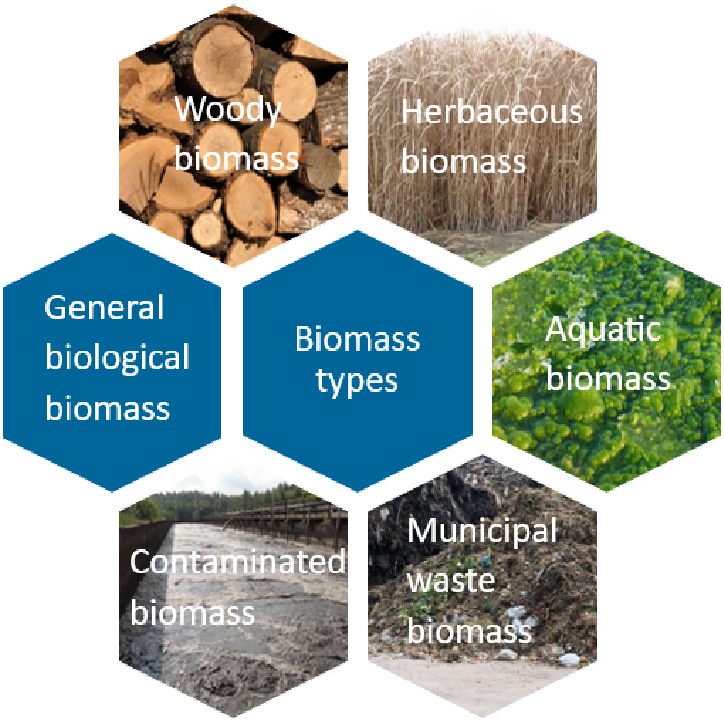


Fig. 2.: *General classification of biomass, adapted from[11]. Picture sources, clockwise, starting with woody biomass: [12][13][14][15][16]*

This thesis is focused around the comparison of woody and non-woody biomass, and their usability in pelletisation and thermochemical gasification, which fall into the categories of woody biomass, as well as herbaceous biomass.[9] Wood and woody biomass includes all biomass with comparative properties of hardness, growth time and chemical composition, typically this includes good mechanical strength, relatively high lignin contents between 20 and 30 wt% and long growth times of several years or even decades.[17] From a supply-perspective, both the primary product (i.e. the trunk wood) and residues such as twigs or bark are included, these residues may be supplied directly from the timber industry, or from secondary sources, such as landscaping residues, drift wood, orchards or furniture industry. In general woody biomass is mostly utilized in a cascading process, meaning it is not primarily grown as fuel, but only used as such once it outlived its usefulness in the original purpose.[18]
Non-woody biomass originates in the agrarian industry, it has comparatively lower mechanical strength, lower lignin contents of only 15 to 20 wt% and much shorter growth periods of only 1-2 years.[18]. Non-woody biomass is categorised as either energy crops, which are purposefully grown to be used as fuel, and residues/wastes where secondary or tertiary products are utilised as fuel in the final step of cascading use. Typical examples for energy crops are miscanthus, switchgrass or reed canary grass, while typical examples for secondary or tertiary

non woody-biomass are wheat- and corn straw , nut-shells or corn cobs.[19]

For this thesis, 5 types of biomass are particularly interesting:

- Hay
  Hay consists of field grasses and herbs, which are dried after harvesting. The chemical composition depends on the time of harvest, the exact plant species present and the type and amount of fertilizer used. Hay is usually dried towards a target moisture content of 15 %. It contains a lot of nutrients and is therefore mostly used to feed livestock. It belongs to the herbaceous biomass category.[20] [19]

- Straw
  Straw is the fibrous remnant of grains which remains after threshing. It usually has a yellowish colour. The chemical composition is substantially different from the actual grain seed utilized for human and livestock food production. One of the main users of straw is mulching, where it is cut and incorporated into the soil. Furthermore, straw is used as bedding material in stables. It belongs to the herbaceous biomass category.[20] [19]

- Corn cobs
  Corn is an annual plant with high starch contents of up to 65 %, which makes it particularly interesting for the production of ethanol. For this, mostly the grains are used, while for the use as fuel, the whole plants can be utilized. In this thesis, the granulated corn cobs without straw or grains are utilized. [19]

- Miscanthus
  Miscanthus, especially *Miscanthus x giganteus* is a perennial type of grass. It is sterile, therefore cloning is required for reproduction. The primary use of miscanthus is as an energy crop, due to its relatively advantageous fuel properties, which include relatively low ash, potassium (K), nitrogen (N) and chlorine (Cl) contents, as well as a generally low moisture content. Those contents can be further modified through type and amount of fertilizer, watering and time of harvest. While miscanthus generally prefers warmer climates, it can still be utilized very well in colder climates, provided that a consistent availability of water is ensured. It belongs to the herbaceous biomass category. [19]

- Beech wood
  Beech is a deciduous tree which can be commonly found in western and central Europe. It is a hardwood which prefers soil with medium wetness and dislikes both waterlogged and dry soil. Overall it is considered a very resilient type of plant, both in regards to insects and storms. Economically speaking, beech wood is one of the most important types of wood, with over two thirds of the annual deciduous tree wood produced in Germany being beech. It belongs to the woody biomass category.[21]

2.2. Ash

As defined in [17], ash is the non combusted fuel remains after incineration, this also includes mineral impurities, originating from the fuel harvesting, processing or transport.

2.2.1. Ash content and appearance

Generally speaking, biomass consists of three main elements, Carbon (C), Hydrogen (H) and Oxygen (O), which make up over 90 % of the elemental composition, plus a number of trace elements found in smaller quantities and varying between different biomass types.[22] On a molecular level, the three main elements form the foundation for cellulose, hemicellulose and lignin, those three bio-polymers also contain the majority of the energy content found in biomass.[22][17] Woody biomass has an overall lower ash content compared to non-woody biomass, thus blending woody and non woody biomass,could allow for a lower total ash content in the product.[23]

Biomass has an inherent ash content , which depending on the type of biomass ranges between 0.5 % for high quality woody biomass, and 20 % found in waste materials contaminated with inert materials like rocks or sand. The inherently present and chemically bound ash is usually homogeneously distributed, while so called foreign ash may move within the biomass. During the combustion, volatile types of ash material may change to a gaseous phase, which can be driven out of the gasifier. This is facilitated through higher combustion temperatures and a reducing atmosphere. Additionally, metallic components may turn into so called fly ash, given high enough process temperature. During this process, the metallic components partially oxidise and agglomerate around cores of Calcium oxide (CaO), the size of those fly ash particles is generally $<0.1\,\mu$m. Most of the ash however is simply partially melted and agglomerates into particles of various sizes and size distributions. These particles form the grate-ash and are found in the grate at the bottom of the reactor. This grate ash may cause considerable issues, namely the clogging of the reactor, should it start to melt.[24] It must also be noted that the findings of [24] are referencing combustion plants, it is however assumed that fixed bed gasifiers, which this thesis focuses on, lead to comparable results.

2.2.2. Ash composition, formation and effects

On an elemental level, the most important ash contents are alkaline earth metals especially calcium or magnesium, silicon, alkalis, especially potassium and sodium, phosphorus, sulfur and metallic components, in particular iron and aluminium. Hereby, potassium is the critical element, as it can form various products with negative effects at all process relevant temperatures. This includes potassium salts like KCl, K_2SO_4 or K_2CO_3 which can cause fouling on downstream heat exchanger surfaces due to their low DT of only about 770 °C, potassium silicates or potassium-aluminium silicates which may lead to ash sintering and bed agglomeration, as well as potassium phosphates which may melt the biomass during the process and result in slagging or ash sintering. A further problem is the formation of aerosols and fines, which may decrease the efficiency of energy conversion.[25]

Table 1 gives an overview over typical ash contents of various biomass types. The values depicted are only approximations however, since harvest time, age, soil composition and other factors may influence the exact values. [26]

Table 1.: *Overview of chemical composition and ash content of relevant biomass types*

	Ash	C	H	N	S	Cl	
Type of biomass	wt% dry mass						Source
Hay	9.4	46.3	5.2	2.4	0.2	0.8	[27]
Straw	5	42.9	5.5	0.6	0.3	0.4	[28]
Miscanthus	3	47.9	5.5	0.2	0.03	0.04	[29]
Corn cobs	1.2	47.8	5.6	0.4	0.01	0.2	[30]
Beech wood	0.6	48.3	5.8	0.2	0.03	n.a.	[31]

The effects of specific elements on both the ash softening behaviour, as well as additional effects on ash and process, according to [6] are shown in table 2. Hereby, K and Si stand out as specifically negative, while Mg is the only element that influence ash softening behavior positively. Furthermore, K, S and Cl all facilitate corrosion within the reactor.

Table 2.: *Effects of elements on ash softening behavior[6]*

Element	Effects on ash softening	Other effects
K	Negative	High temperature corrosion
P	n.a.	Viable for fertilizer
Ca	n.a.	Decreases SO_2 in exhaust
Mg	Positive	n.a.
Si	Negative	Decreases aerosol formation
S	n.a.	SO_2 emission; corrosion
Cl	n.a.	Corrosion; aerosol formaion

The exact composition varies between different biomass types, woody biomass usually has higher calcium and magnesium contents, whereas non-woody biomass contains more potassium, sodium and silicon. Waste products and coal on the other hand contain considerable amounts of metallic components. [32] [33] In addition to the main components, secondary components such as of As, Cd, Co, Cu, Hg, Mn, Mo, Ni, Pb, Sb, V and Zn can also be found within biomass ash in small quantities. The actual appearance of the different elements is shown in table 3. Hereby, calcium can be present in a multitude of different forms, while silicon only takes one form and potassium only two.[34]

Table 3.: *Appearance of inorganic compounds within biomass [33]*

Element	Ionised salt	Compound	Organic form	Mineral
K	Potassium nitrate	KNO_3		
	Potassium chloride	KCl		
Ca	Calcium nitrate	$Ca(NO_3)_2$	Calcium pectin	Calcium oxalate
	Calcium chloride	$CaCl_2$		
	Calcium phosphate	$CaHPO_4$		
		$Ca(H_2PO_4)_2$		
		$Ca_3(PO_4)_2$		
Si	Amorphous silicon	SiO_2		Phytoith
				Quartz

During the combustion process present potassium components change into liquid or gaseous phase once the temperature rises beyond 770 °C. These liquids and gases either become fly ash or react with the metallic components in the bed to form various silicates and aluminium silicates, such as $K_2Si_2O_5$ or $KAlSiO_4$. Metallic components which have not yet reacted with potassium components usually form oxides, such as MgO or Fe_2O_3. Calcium based components have comparatively high melting points which results in them remaining in solid form, they do however react with sulfates, carbonates, silicon and phosphorous. The presence of phosphorous in particular leads to the formation of hydroxyapatite $Ca_5(PO_4)_3(OH)$, a common component of biomass ash.[33]

When looking at the effects of ash components, nitrogen, potassium, sodium, magnesium, calcium and silicon all influence ash softening and meting behaviors. Nitrogen may lead to NOx and N_2O emissions, while all other common components can lead to particle emissions. Ash containing potassium, magnesium calcium, phosphorous or silicon may be utilized in different processes, such as fertilizer. High temperature corrosion can occur when notable potassium, chlorine or sulfur components are present. Ash may also serve as a binder for other pollutants, magnesium, sodium, calcium and phosphorous-based compounds have shown positive results in that regard.[17]

According to Nunes et al.[24] SiO_2 and CaO are the most common chemical compounds found in biomass, table 4 shows however, that K_2O is also commonly found in larger quantities. It has to be noted that composition varies greatly between different plant types, generally speaking though, woody and non-woody biomass have more similarities within their respective class than when compared to each other.[24]

Table 4.: *Chemical composition of biomass ash[6]*

	Ash composition in wt%				
Chemicals/Fuel	Hay	Straw	Corn cobs	Miscanthus	Beech wood
Sources	[35],[6]	[6]	[36], [34],[6]	[35], [37], [38],[6]	[39]
SiO_2	43.69	49.02	39.43	59.76	14.62
CaO	15.24	8.75	3.96	6.72	31.06
K_2O	20.32	23.3	37.3	18.53	18.80
P_2O_5	6.1	3.84	4.36	3.42	12.87
Al_2O_3	6	1.09	5.24	0.62	0.76
MgO	4.47	2.34	3.57	4.37	6.87
Fe_2O_3	2.44	0.85	1.49	0.71	0.65
SO_3	1.42	3.38	2.58	1.7	1.09
Na_2O	n.a.	2	0.75	0.41	0.40
TiO_2	n.a.	0.28	0.32	0.4	0.06
Cl	0.32	5.03	0.97	3.27	n.a.
MnO	n.a.	0.13	0.09	0.1	6.35

The various chemical compounds shown in table 4 can cause disadvantageous effects on the reactor. One notable example is the formation of KCl, which causes corrosion. Additives can help to combat those risks.[26]

2.2.3. Experimental ash melting behaviour

Depending on the ash-composition ash starts to liquefy at certain temperatures. This can occur with two different mechanisms, the melting dissolution and the softening-melting.
Melting dissolution mostly occurs in ashes based on biomass-fuel. At first, a low viscosity molten ash form at lower temperatures, which can dissolve contents with higher melting points. [34]
Softening-melting is prevalent in coal based fuels, it consists of a prolonged softening period followed by the formation of high-temperature melts with low viscosity, which in turn slows down further melting.[34]

Softening and subsequent melting takes place in multiple steps. [34], [40] and [6] summarised those steps, which are standardised by the EN ISO 21404[7] and DIN 51730[8] standardisation sets. A visualisation of the different steps for each standardisation is shown in fig.4 and fig.5. The DIN 51730[8] is used for solid fuels, while the EN ISO 21404[7] is used for biogen fuels. While both can be considered relevant for this thesis, the newer EN ISO

21404[7] is primarily used.
For both standards, a cylindrical sample consisting of at least 2 g ash is fabricated. Fig.3 shows such a sample cylinder. [41][6]

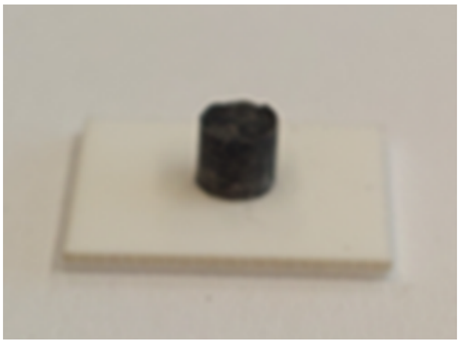


Fig. 3.: *Cylindrical sample, produced according to [41]*

Definition according to DIN 51730[8]

For the EN ISO 51730 standardisation, the definitions, as depicted in fig.4 are as follows:

- OS (Original sample): The OS is the shape and size of the sample before the start of the heating program.
- DT (Deformation temperature): The DT is defined as the temperature at which first signs of rounding off at the edges are visible, furthermore swelling also counts as deformation. In the DIN 51730[8] standardisation the required temperature to reach the deformation stage (DT) is defined as the point where a form change of 15 % is reached. The form change factor is defined as $F = a/b$, where a is the circumference of the sample and b is the circumference of a semicircle with the same area as the shadow of the sample.
- ST (Spherical temperature): In cubic and cylindrical samples, the ST is the temperature at which the edges of the sample become round, while no change in height takes place.
- HT (Hemispherical temperature): The HT is defined as the point where the sample takes on an approximately hemispherical shape, while simultaneously the height equals half the diameter of the base area of the sample.
- FT (Flow temperature): The FT is the temperature at which the sample forms a layer on the sample carrier, where the current height is on third of the height at the hemispherical temperature.

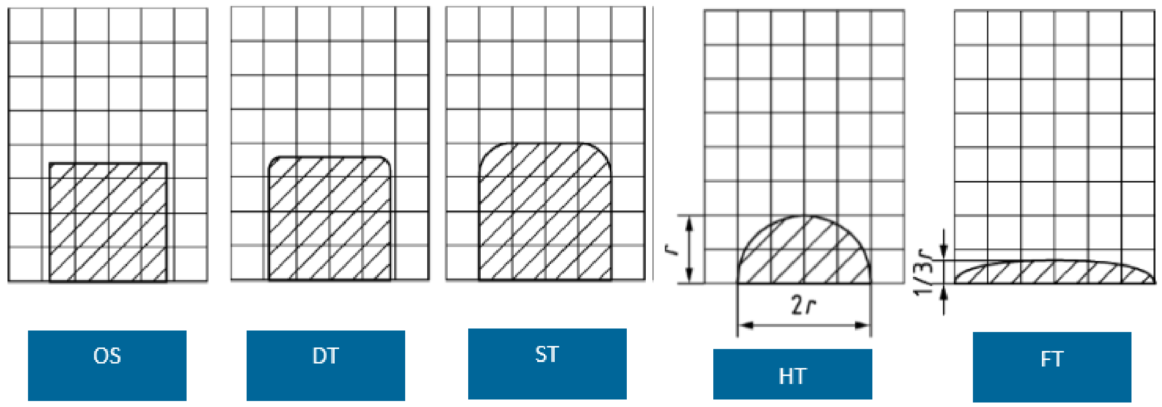


Fig. 4.: *Characteristic temperature of ash melting according to EN ISO DIN 51730, adapted from [6]*

Definition according to DIN EN ISO 21404[7]

For the EN ISO 21404 standardisation, the definitions, as depicted in fig.5 are as follows:

- OS (Original sample): The OS is the shape and size of the sample before the start of the heating program.
- SST (Shrinkage starting temperature): The SST is defined as the temperature at which the area of the sample is smaller than 95 % of the original sample area at 550 °C.
- DT (Deformation temperature): The DT is defined as the temperature at which first signs of rounding off at the edges are visible, furthermore swelling also counts as deformation.
- HT (Hemispherical temperature): The HT is defined as the point where the sample takes on an approximately hemispherical shape, while simultaneously the height equals half the diameter of the base area of the sample.
- FT (Flow temperature): The FT is the temperature at which the sample forms a layer on the sample carrier, where the height is half that of the sample at the hemispherical temperature.

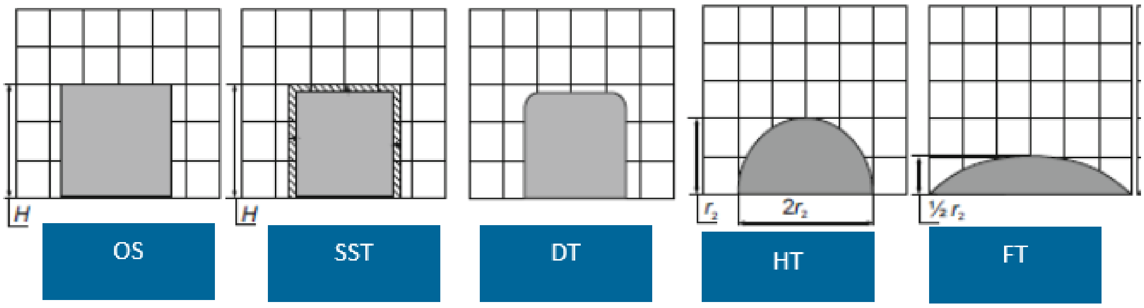


Fig. 5.: *Characteristic temperature of ash melting according to EN ISO DIN 21404, adapted from [6]*

2.2.4. Estimation of ash melting behaviours

There are different ways to evaluate the ash melting behaviours of fuel before actual experimental set up. These could theoretically serve as a powerful tool to save costs and efforts, if they are properly calibrated to yield accurate results. Currently these estimations are only tested for combustion processes, although data suggests they can still be used for gasification processes as an approximation. Lessig[6] and Sommersacher[42] summarised the results regarding those possible approximations.

One possible way to asses the ash melting behaviour is the use of fuel indices. Depending on the elemental composition of the fuel, four different indices may be used.It is important to note that those indices all indicate the SST, it is however not guaranteed that the SST is the defining point for potential reactor clogging, but merely the first temperature dependant change in ash properties. It is assumed that the DT is the more important breaking point, since it marks the point of changes in shape.

The basic index is used for fuel mostly defined by Silica, Calcium and Magnesium.

$$\frac{\mathrm{Si}}{\mathrm{Ca}+\mathrm{Mg}} \text{in mol/mol} \tag{1}$$

Generally speaking, when this index takes on values above 1, the SST drops below 1100 °C. For non-woody biomass typical index values are around 2.5, which indicates that the DT is lower than 1000 °C.

When phosphorous and potassium are present, the index is extended to:

$$\frac{\mathrm{Si}+\mathrm{P}+\mathrm{K}}{\mathrm{Ca}+\mathrm{Mg}} \text{in mol/mol} \tag{2}$$

Equal to the first depicted index, values above 1 equal a SST below 1100 °C. Typical values for non-woody biomass are even higher, usually ranging about 5.

Finally, when aluminium is present the index gets extended to:

$$\frac{\mathrm{Si} + \mathrm{P} + \mathrm{K}}{\mathrm{Ca} + \mathrm{Mg} + \mathrm{Al}} \text{in mol/mol} \tag{3}$$

Values lower than 1 indicate ash softening only starts above 1200 °C. For this thesis, the aluminium index is used primarily, due to all considered elements are expected to be present within the ash, as shown in table4. When the index value ranges between 2 and 3, the expected SST is around 1100 °C. Due to the specifications of the Glock gasifier, temperatures of up to 1200 °C may be reached in parts of the reactor, thus an ash softening temperature of more than 1200 °C marks the target. This in turn means the target index values are lower than 1.[6]

- Indices lower than 1 indicate SST above 1200 °C, they are marked in Green
- Indices between 1 and 2 indicate SST between 1100 °C and 1200 °C, they are marked in Yellow
- Indices between 2 and 3 indicate SST around 1100 °C, they are marked in Orange
- Indices above 3 indicate SST lower than 1100 °C, they are marked in Red

The calculated results are displayed in table 5.

Table 5.: *Ash softening indices[6]*

Index	Additive	Index			
Fuel	wt%	Hay	Straw	Corn cobs	Miscanthus
$\frac{Si}{Ca+Mg}$	None	1.9	3.81	4.12	4.36
$\frac{Si+P+K}{Ca+Mg}$	None	3.25	6.38	9.48	6.29
$\frac{Si+P+K}{Ca+Mg+Al}$	None	2.49	5.8	5.76	5.98
——〃——	Kaolin				
——〃——	2.5	2.08	3.65	3.77	3.7
——〃——	5	1.85	2.84	2.95	2.95
——〃——	Dolomite				
——〃——	2.5	1.63	2.73	2.87	2.86
——〃——	5	1.22	1.8	2.21	1.89
——〃——	Lime				
——〃——	2.5	1.66	2.82	2.97	2.96
——〃——	5	1.25	1.87	2.0	1.97
——〃——	7.5	1.00	1.40	1.51	1.47
——〃——	Beech wood				
——〃——	33	2.03	4.24	4.21	4.36
——〃——	66	1.57	2.68	2.66	2.74

The calculated data suggests that it is almost impossible to reach an SST above 1200 °C with the considered additives. It however confirmed and well known that wood is a viable fuel that can be utilized in gasifiers without the risk of clogging due to ash softening. Because of that, this thesis will focus mostly on the DT for the analysis of the ash softening behaviour, since it is assumed that the shrinking is not the primary factor for clogging within the gasifier.

2.3. Mechanical preconditioning and pelleting

2.3.1. Pellet properties

The properties of pellets can be categorised in chemical and physical properties. Chemical properties depend on the raw material, while physical properties are defined by the pelletisation process. According to [17] and [43] the physical properties are physical dimensions (length, diameter), pellet-, raw- and bulk-density, energy-density, grain size distribution, moisture content (MC), durability and calorific value.

Tolerable dimensions of pellets are classified by DIN EN ISO 17225-2[44]. There are 5 classes, D06, D08, D10, D12 and D25. The number indicates the maximum diameter in millimeters, with a tolerance of 1 mm. For D06, D08 and D10, the length must be between 3.15 and 40 mm, for D12 between 3.15 and 50 mm and for D25 between 10 and 50 mm. According to DIN EN ISO 17225-2[44], only D06 and D08 are both eligible for industrial and private usage, while D10, D12 and D25 are only eligible for industrial use. In addition to length and diameter, volume, geometrical form and surface properties are also included in this category. For the use of pellets, only cylindrical forms are relevant.[17][43][44]

Raw and pellet density classify the density of the raw material and the pellet respectively. In general, woody biomass has higher density compared to non-woody biomass. The density influences the transport behavior in pneumatic transport, as well as the bulk density.[17][43][44]

The bulk density is defined as the fuel mass divided by the volume of the storage vessel, it is important for the storage and transport behaviour.[17][43][44]

Energy density is defined as the energy unit per either mass or volume. The energy units are hereby based on the lower heating value at a predefined water content.[17][43][44]

To classify the various sizes within the bulk, the grain size distribution is used. The pellets are categorised into a main component (at least 60 wt%), fine component (diameter <3.15 mm) and a coarse component (max length, defined by DIN EN ISO 17225-2[44]). Additionally, the maximum length and cross-section area are defined. If the share of fine components is too high, the fuel may transforms from a homogeneous mixture to a heterogeneous mixture, while an over abundance of large parts facilitates bridge formation and blockages, which in turn reduces throughput.[17][43][44]

There are two different definitions for the moisture content, one usually used for non-woody biomass and one for woody biomass:
In case of non-woody biomass, the moisture content is defined as the water content within the wet biomass, where the wet biomass is the sum of the dry biomass and the water content.

It is measured in %. [17]

$$w = \frac{m_W}{m_B + m_W} \cdot 100 \qquad (4)$$

For woody biomass, the water content in relation to the dry mass is used instead, with the unit being again %. Due to this definition, values above 100 % may occur. [17]

$$u = \frac{m_W}{m_B} \cdot 100 \qquad (5)$$

Durability, sometimes also called mechanical strength, describes the internal cohesion of particles within a dense body like a pellet or a briquette. Therefore the durability directly relates to dust formation when frictional forces are applied during transport. According to DIN EN ISO 17225[44], a maximum mass loss of 2.5 % in form of dust is tolerable. Potential risks are dust abrasion and pollution within the vicinity, as well as the potential risk of dust explosions. The durability is influenced by the choice of raw material as well as the pelletisation process parameters.[17][43]

There are 2 different calorific values, the net calorific value and the gross calorific value. The net calorific value is defined as the potential release of energy in full oxidation, without the inclusion of water evaporation heat. It is defined by the DIN EN 14918[45] standardisation and is also known as lower heating value.
In contrary, the gross heating value or higher heating value includes the use of the evaporation heat. For industrial uses, the lower heating value bears more importance.[6][17]

2.3.2. Process

The purpose of mechanical preconditioning is the adaptation of fuel-properties to be advantageous during the combustion process. Those properties include size, density, grain size distribution[46], as well as the proportion of fines and the moisture content[47]. As described by [46], there different categories of mechanical preconditioning, some examples are fragmentation, pelleting and torrefication.

Fragmentation is defined as a process of size reduction of a material compound through use of mechanical forces. The classification of fragmentation equipment is done either through product size (crusher/mills) or fragmentation process (compression/tensile stress, impact, cutting, decompression). The choice of fragmentation used depends on the material properties, material size and target size. For fibrous, non woody materials, fragmentation trough cutting is preferred, while crushing and milling is used for other materials.[46]

Figure 6 shows an exemplary depiction of the structure and functionality of the most important fragmentation machine types. Jaw and cone crushers decrease the size of the material through the application of pressure.

Hammer and impact mills accelerate the material and/or smash it, the impact causes it to break apart. Additionally, shear forces help with the fragmentation process. In hammer mills, the impact force is directly applied through the hammers hitting the feed material, while in impact mills the cleats simply accelerate the material, the actual impacts happens at the baffle plates.[46]

Cutting mills fragment the feed material, as the name suggest, through the use of blades which cut the material into smaller pieces. They are mostly used for fibrous materials with lower density, where hammer and impact mills sometimes struggle. The main disadvantage is the susceptibility to hard foreign bodies such as rocks, as they may dull or nick the blades. To prevent this, proper pre-treatment of the feed material is necessary.[46]

Grinding mills, such as ball mills or vertical rollers function through the use of pressure. Ball mills utilize loose balls within a rotating drum, while in vertical roller mills, a rotating grinding roll is fixed in place above a roller plate, the material is milled in the contact zone between the two. Grinding mills are especially used to mill packed materials such as pellets or briquettes. The advantages of such mills are low wear and high throughput, under the condition that no hard impurities such as metals are present. The main disadvantages are the size of the machinery, high noise pollution and the risk of sparks, which can be prevented through the use of inert atmospheres.[46]

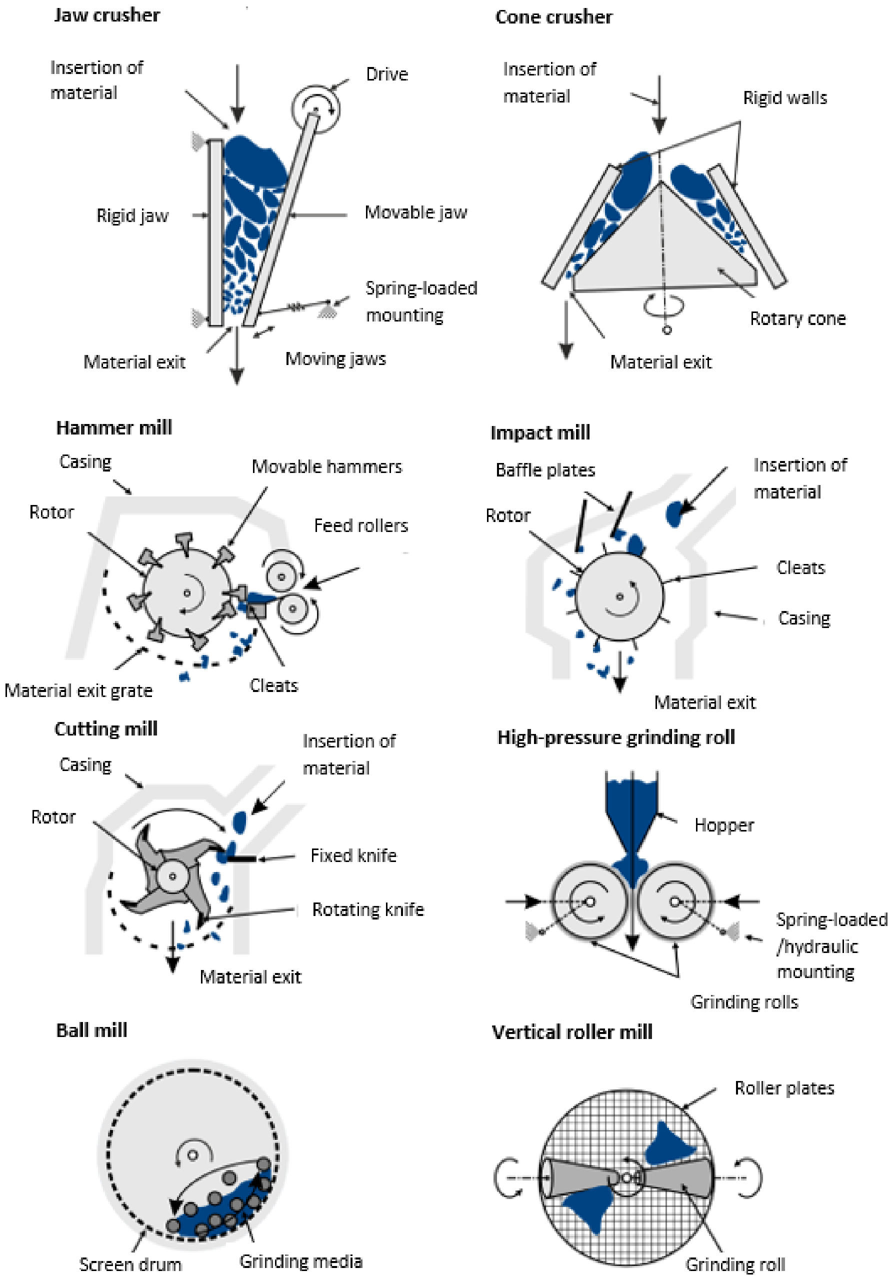


Fig. 6.: *Drawings of various milling and fragmentation technologies, taken from[48]*

Pelleting is the most expensive and extensive of the mechanical precondition of biomass fuels, but pellets offer a variety of advantages over other biomass fuels. The advantages, as described in[46] are:

- High energy density and homogeneity
- Favorable flow and dosing characteristics
- Low moisture content and storage stability
- Easy incorporation of additives

- Lower proportion of fines when compared to other biomass material

Overall, pellets are preferable to loose fuel, due to the aforementioned properties, the actual quality of pellets may vary however. Important influence factors are the choice and quality of the feed material, potential additives, as well as the influence of pre and post treatment. Additionally process parameters can noticeably influence the final quality.[23]

Figure 7 shows the exemplary pellet production process of four different raw and waste biomass materials. It has to be noted that most of the mechanical preconditioning is done before the drying step, furthermore the necessary steps vary considerably between the different biomass types.

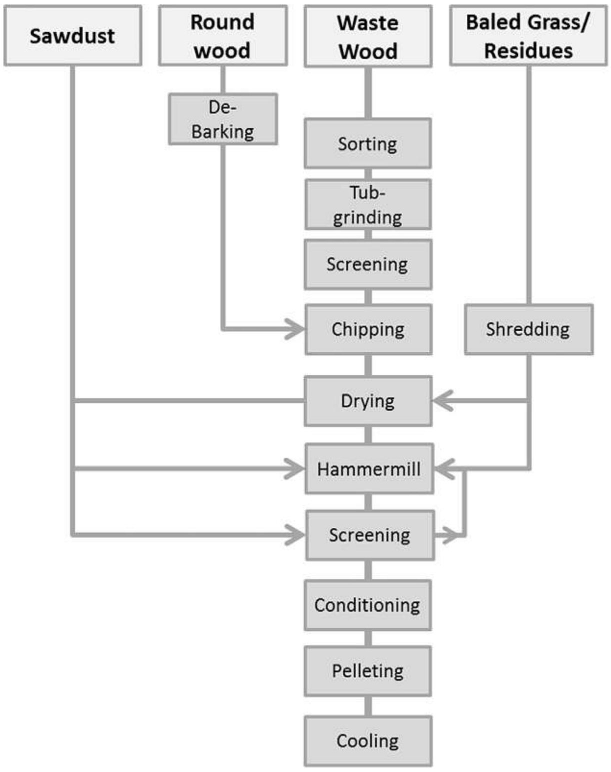


Fig. 7.: *Pelleting procedure, taken from [47]*

According to Whittaker et al.[47] the moisture content of fresh woody biomass is usually around 50-55 %, while non woody biomass like miscanthus usually have a moisture content of about 20 % although depending on the season of harvest, this may vary. For ideal pelleting the desired moisture content is between 8 and 12 %, thus drying often becomes necessary. The drying step is generally done before milling the feed-stock, as additional moisture content may cause unwanted agglomeration. Typical drying technologies include hot air, desiccation and vacuum drying. Longer drying time leads to an increased loss of volatile contents. [47]

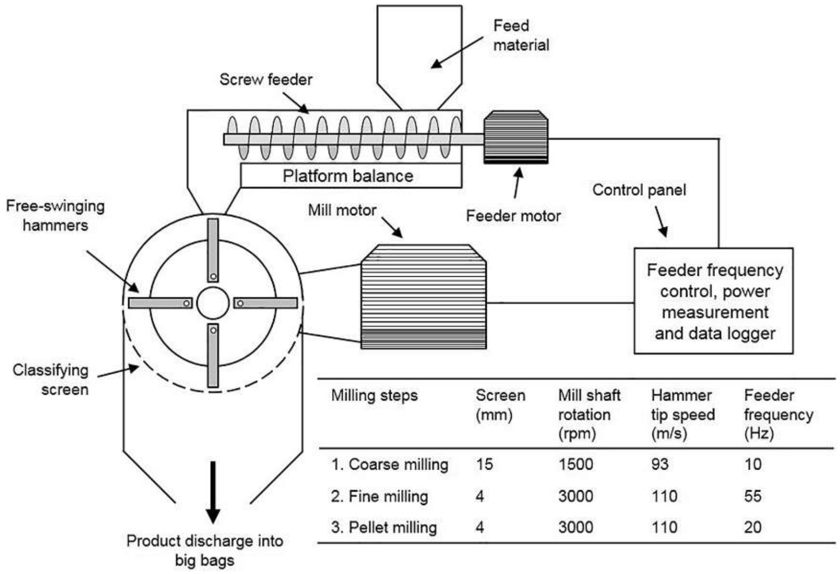
Torrefaction is a special form of thermal pre-treatment to decrease volatiles and decrease moisture content, and is usually done at a temperature range of 200-300 °C in an inert atmosphere. The main process is the decomposition of hemicellulose. Torrefaction can reduce the total mass of the feed by up to 30 % while only losing about 10 % of the total energy content. Additionally this process increases the density of the biomass, which can lead to a more challenging pelletisation process afterwards, although the also higher energy density means that torrefied pellets usually have a comparatively higher quality.[49]

The particle size is a crucial influence factor for a number of pellet properties, in particular hardness, total surface area, pore size and bonding between particles. For the production of pellets hammer mills are the generally used technology. Target properties during the milling process are a size larger than 3.15 mm but no larger than 8 mm, a homogeneous particle size and grade of conditioning, as well as a coarse structure to improve combustibility, even though a coarse structure tends to reduce pellet durability.[47]
Additionally, a size reduction increases bulk density and biomass flow, as well as inter particle bonding and a reduction of specific energy consumption during the later pelletisation process.[49]

Before the pelletisation process the biomass is conditioned. This serves the purpose of a final adjustment of the water content, homogenisation of the biomass and the activation and improvement of binding properties. Additionally, additives are incorporated in this step. [46]. Steam explosion is one form of conditioning in which hot steam at around 200-260 °C is inserted in to the biomass for up to 10 min, afterwards a decompression is conducted. This allows for improved binding properties since it gives better access to bio-polymers while not requiring pressurised reactors. Pellets produced using this process generally have a higher pellet density.[49]

Pellets are defined as processed bio-fuels in usually cylindrical shape with the properties of a bulk material. They can be produced with or without the utilisation of additional binders. Typical diameters range fro 6 to 8 mm, while the spread-with ranges from 5 to 30 mm, as defined be the DIN ISO 16559 standardisation.[46][50]

The standard device for the production of pellets is the so called pan grinder mill. Figure 8 shows an exemplary pan grinder mill, the preconditioned feed-stock is fed into the mill through a screw. The actual mill consists of a pan matrix and a rotating grinding wheel. During the milling process, a layer pressed feed-stock forms through the rotation of the grinding wheel. Once sufficient density is reached, the compressed mass is pressed through holes in the pan matrix and cut into predefined length beneath it. This process releases considerable heat energy which further dries the pellets, as well as facilitating agglomeration.[46]



Milling steps	Screen (mm)	Mill shaft rotation (rpm)	Hammer tip speed (m/s)	Feeder frequency (Hz)
1. Coarse milling	15	1500	93	10
2. Fine milling	4	3000	110	55
3. Pellet milling	4	3000	110	20

Fig. 8.: *Drawing of a pan grinder mill, taken from [51]*

2.4. Thermochemical conversion

The thermochemical conversion describes the chemical changes happening to the fuel material under the influence of temperature and when reacting with gases within the process. One important influencing variable is hereby the amount of air within the process. According to Delalamo et al. [52], this ratio, lambda (λ), is defined as: "The ratio of the gasification air to the total stoichiometric air for complete combustion of the fuel input." [52]. Lambda values between zero and one, and thus a lack of air are called a rich mixture, a value of one indicates a stochimetric mix, and mixtures with a lambda value of more than one are called a lean mixture. Full oxidation happens at lambda values of one and greater.[53]

Thermochemical conversion of biomass takes place in four steps at different temperature ranges and different air to fuel ratios. The process is schematically pictured in fig.9. Both [54] and [17] describe the thermochemical conversion of biomass:

The first step is drying, which happens up to a temperature of 100 °C, after that the biomass is pre-heated to a temperate of 150 °C for the next process step. During the drying, surface and capillary water are driven out of the fuel, while no chemical changes take place. The degree of drying does however influence the necessary temperatures for the second step. Drying marks the most energy-intensive step of thermochemical conversion.

Depending on the actual pyrolysis temperature, the product composition varies. At lower temperature around 300 °C coke and gaseous products predominate. As the temperature rises the amount of tar increases, while the amount of coke decreases, up to approximately 650 °C where mostly gaseous products remain. Tar can occur in liquid (paralysis-oil) or gaseous form, with the actual composition depending on the fuel-composition and process parameters. The gaseous products are mostly carbon monoxide (CO), carbon dioxide (CO_2), methane (CH_4), as well as higher carbohydrates and water. [55] According to Li et. al. [56], tars are defined as organic impurities and particles, which can be found in the form of polynuclear aromatic hydrocarbons with a high molecular weight. During further process steps, present tars may condense or polymerize into other, more complex structures. Both tars in their original form and their chemically transformed products have negative impacts on the process, as they often cause clogging and choking, which can decrease the efficiency of the process and in turn increase operational costs. The removal of tars is costly and complex, relying on thermal, catalytic and physical processes. If possible, avoiding the formation of tars altogether is preferable.[56]

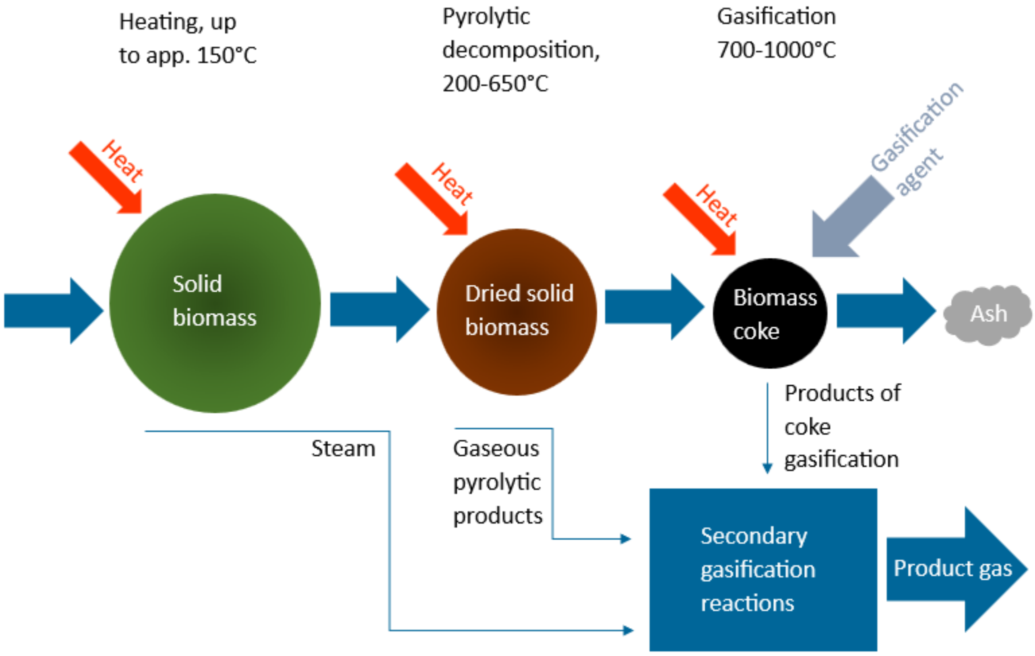


Fig. 9.: *Thermochemical conversion of biomass, adapted from[17]*

The third step of thermochemical conversion is gasification, which takes place between 700 °C and 1000 °C. In this step, the products of the pyrolysis are converted into combustible gases. The addition of a gasification agent is necessary during this step, typical examples are: air, O_2, H_2O or CO_2. To avoid total oxidation, substoichiometric conditions are necessary. Typical ratios between the fuel and the gasification agent, i.e. the lambda value, range from 0.3 to 0.4. During this step a multitude of different reactions take place. While some of the reactions taking place are exothermic, the overall process is endothermic, thus additional energy has to be brought into the process. To source the required additional energy, two process types are utilized. In auto-thermal reactors, some of the fuel is fully oxidized and the released energy, alongside the energy from the exothermic reaction is used to operate the gasification. The alternative are allothermal processes, where the necessary energy is inserted into the process from external sources. The most important of the reactions taking place during gasification are[55][17]:

$$C + CO_2 \longrightarrow 2\,CO \quad \Delta H_{R,25\,^\circ C}{=}172\,\mathrm{kJ/mol} \quad \text{Boudouard reaction [55]} \tag{6}$$

$$C + H_2O \longrightarrow CO + H_2 \quad \Delta H_{R,25\,^\circ C}{=}131\,\mathrm{kJ/mol} \quad \text{Char reforming [55]} \tag{7}$$

$$CO + H_2O \longleftrightarrow CO_2 + H_2 \quad \Delta H_{R,25\,^\circ C}{=}-41\,\mathrm{kJ/mol} \quad \text{Water gas shift reaction [55]} \tag{8}$$

$$C + 2\,H_2 \longrightarrow CH_4 \quad \Delta H_{R,25\,^\circ C}{=}-75\,\mathrm{kJ/mol} \quad \text{Methanisation [55]} \tag{9}$$

The final step of thermochemical conversion is full oxidation, where only CO_2 and H_2O remain as products. To achieve total conversion, the fuel-to gasification agent ratio or lambda value has to be 1 or higher. [32] The chemical reactions taking place during the oxidation step are [17]:

$$C + O_2 \longrightarrow CO_2 \quad \Delta H_{R,25\,^\circ C}{=}-394\,\mathrm{kJ/mol} \quad \text{Char combustion[55]} \tag{10}$$

$$C + \frac{1}{2}\,O_2 \longrightarrow CO \quad \Delta H_{R,25\,^\circ C}{=}-1114\,\mathrm{kJ/mol} \quad \text{Partial Oxidation[55]} \tag{11}$$

$$H_2 + \frac{1}{2}\,O_2 \longrightarrow H_2O \quad \Delta H_{R,25\,^\circ C}{=}-242\,\mathrm{kJ/mol} \quad \text{Hydrogen combustion[55]} \tag{12}$$

2.5. Gasification technology

Gasifiers serve as a technology to industrially utilize the chemical processes described in chapter 2.4. The different reactor types are defined by the fluid design, heat transfer, gasification agent and the quality and composition of the product gas. A visual overview of the main classification and reactor types is shown in fig. 10[55].

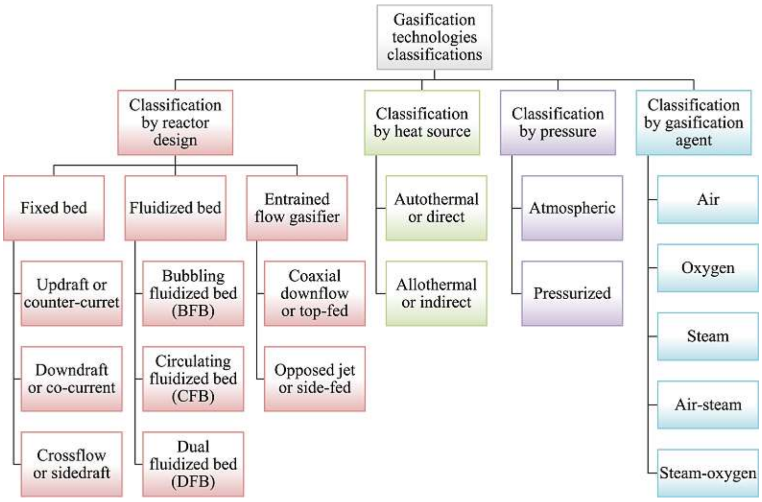


Fig. 10.: *Overview of the different types of gasifiers, taken from[57]*

- The red marked parts in fig. 10 show the three main gasifier types, fixed bed, fluidized bed and entrained flow, as well as their specific sub-types. Here, they are only shown with a single stage, for larger plants it is also possible to utilize multiple gasifiers in cascade or staged use. [58]
- The green part shows the heat source, autothermal means the required energy comes through the process itself, usually through the combustion of some of the inserted fuel, while for allothermal operation heat is brought into the process from outside source, for example excess heat of other nearby processes. [17]
- Purple marks the used pressure, currently atmospheric pressure is predominantly used. [58]
- Cyan depicts the utilized gasification agent. The chemical composition of the product gas is largely defined by the the chosen gasification agent. Oxygen (O_2) and carbon dioxide(CO_2) lead to the formation of carbon monoxide(CO), water to CO and hydrogen (H_2), air to CO and nitrogen (N_2) and H_2 to methane(CH_4). So far, O_2, steam and CO_2 are mainly used.[58]

In fixed bed gasifiers the fuel is put into the reactor from the top and forms a layer or bed.

This bed keeps its structure since either large fuel chunks are used, or the flow speed of the gas is low enough to not cause significant movement of the bed. As the gasification agent flows trough the bed, thermochemical conversion takes place. During the gasification process, the bed material slowly moves form top to bottom due to gravitational forces, as well as the aforementioned thermochemical conversion. Fixed bed gasifiers are further classified by the direction of the gas-flow, for this thesis, two types are further specified: up-draft and down-draft gasifiers.[58]

A simplified flow chart of a combined heat and power plant (CHP), in this case an autothermal fixed bed downstream gasifier, is shown in fig.11. Fuel enters the process through the fuel lock, if necessary a final purification may also take place during this step. The gasification process, which this thesis focuses on, takes place within the reformer, also known as gasifier. The gasification agent goes through the side channel blower into the gasifier, the point of entry is hereby an important distinction between gasifier types. The fuel is gasified, with non-gasifieable components being turned into ash. If those ashes exhibit low SST, ash softening and subsequent clogging of the gasifier can take place. After gasification the product gas, still loaded with ash is cooled down, with the excess heat being utilized elsewhere. In case of failure, a flare combusts excess gas the prevent damage the the plant. After cooling, the ashes are filtered out of the product gas and the now clean gas can be used in the main CHP unit to generate heat and power. The ashes are collected and depending on their contents, further utilized. The gasifier types mainly used in such plants are fixed bed gasifiers, usually in downdraft configuration. [59]

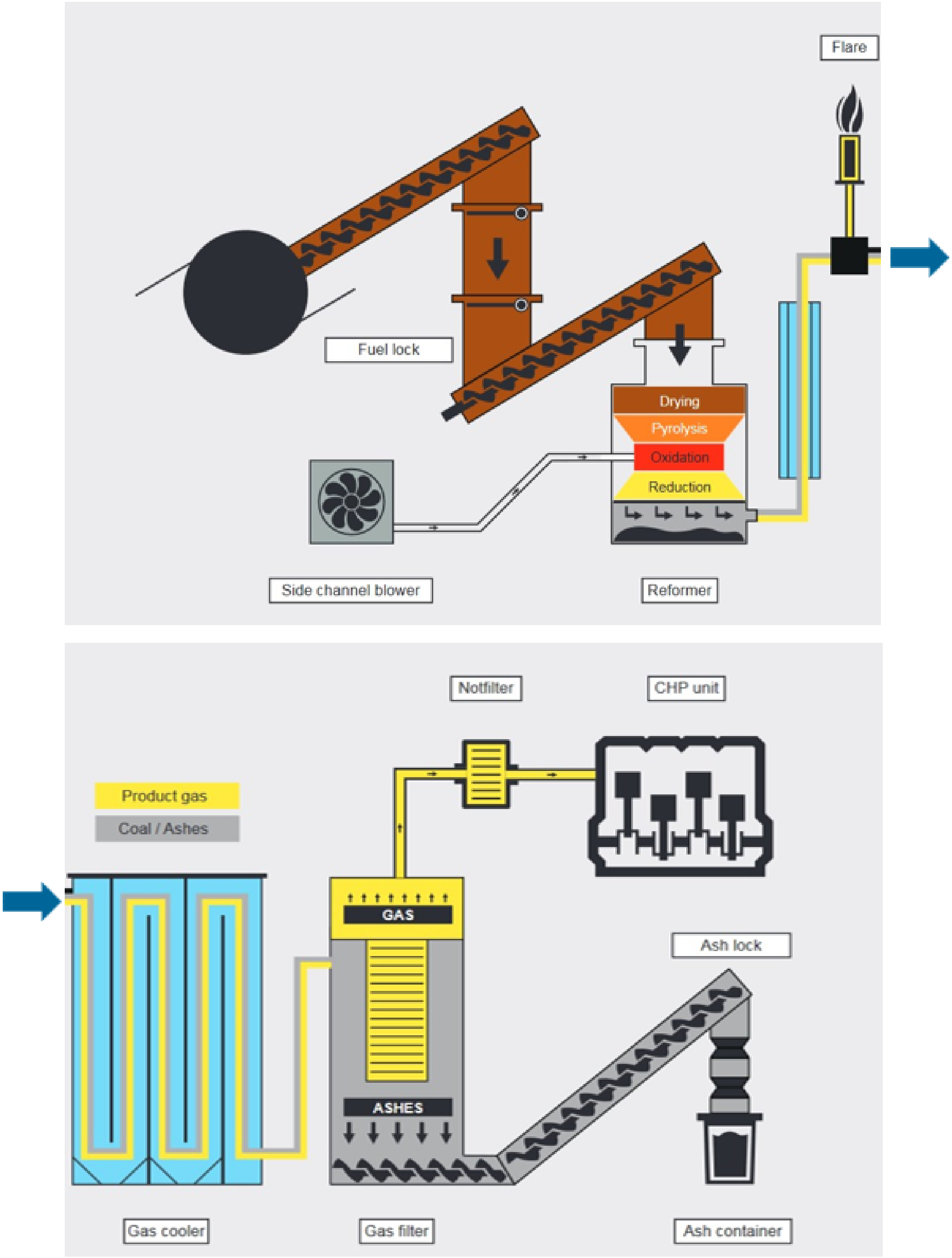


Fig. 11.: *Exemplary flow diagram of a CHP plant, taken from [59]*

A general depiction of up- (a) and downdraft (b) fixed bed gasifiers is shown in fig.12. As the name implies, and contrary to fluidized bed gasifiers, the air flow does not cause notable upward movement of the bed material in fixed bed gasifiers. The gasification process takes place in 5 steps, 4 of which happen inside the reactor with the final step, purification, taking place afterwards.[55]

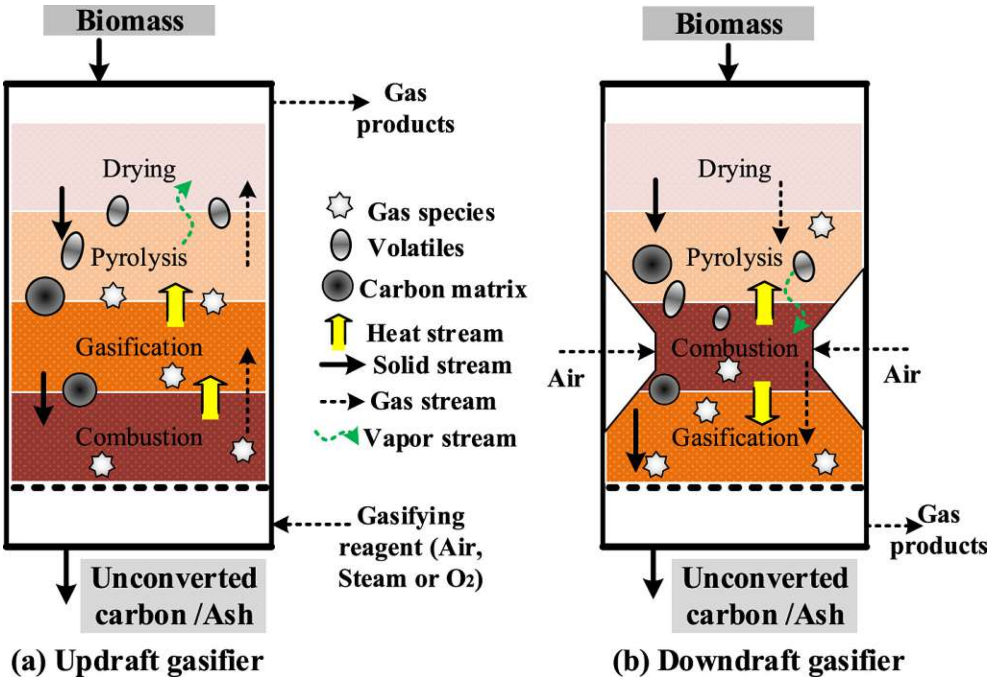


Fig. 12.: *Up- and downdraft fixed bed gasifiers, taken from [60]*

In the first step, the feed material enters the reactor from the top and gets into the drying zone. Under temperatures of up to 150 °C, surface and capillary bound water is evaporated.[55]

The feed material sinks down into the pyrolysis zone, where the temperature ranges from 200 to 650 °C. During this process step, volatiles and gaseous pyrolysis products are driven out of the biomass. During pyrolysis, the matrix of carbon materials is decomposed by cracking. The products of this process can take on mixtures of different physical states. The exact fraction depend on the type of reactor used, for fixed bed gasifiers, the typical solid product content ranges between 20 and 25 wt%, while the liquid fraction, which mostly contains tars, ranges between 10 and 20 wt% for updraft gasifiers and about 1 wt% in downdraft gasifiers. The generalised overall reaction during pyrolysis is[55][6]:

$$\text{Biomass} \longrightarrow \text{H}_2 + \text{CO} + \text{CO}_2 + \text{CH}_4 + \text{H}_2\text{O}\,(\text{g}) + \text{Tar} + \text{Char} \qquad (13)$$

Due to the overall endothermic nature of the process additional energy must be added. In allothermal processes, this energy purely comes from outside sources, while autothermal processes utilize the oxidation step to provide most of the needed energy. During the process the excess air number λ is between 0 and 1, which means only some of the fuel can be fully and exothermic oxidized.[55][6]

Depending on the reactor type, the next zone is reduction/gasification or the combustion/oxidation zone. In the oxidation zone, fuel is combusted and forms carbon monoxide (CO_2), water (H_2O), coke and ash out of non-combustible contents. The highest temperatures, up to 1200 °C occur in this zone. Therefore, ash softening and melting also takes place within the oxidation zone. The results of ash softening and melting are the formation clumps on the reactors inner surface which may clog the system, as the must be removed manually due to their high hardness. [58]

In the reduction zone, temperatures of up to 1000 °C are reached. In this zone, long chained organic compounds are converted into shorter chained compounds, if the process flow allows the to move through it, which is the case in downdraft-gasifiers. Additionally CO, CH_4 and H_2 all form within the reduction zone. [58]

In up-draft gasifiers, the gasification medium flows in the opposite direction of the fuel. This opposite movement leads to the different steps of thermochemical conversion taking place in clearly separated zones. Hereby the hottest zone, where oxidation takes place is at the bottom of the reactor. Main advantages of up-draft gasifiers are the high efficiency, as well as the low exit-temperature, which leads to a lower content content of alkali-metals. Furthermore, such gasifiers are of rather simple construction, which can be utilized for a large variety of fuel-types. Main disadvantages are the higher rate of tar formation and the vulnerability to ash melting and subsequent clumping. Furthermore, up-draft gasifiers have up 100 times the tar content of down draft gasifiers. The reason for this is the low product gas temperature, since the tars don not move through the oxidation zone, which lead to the tars not being thermochemically converted.[58]

In down-draft gasifiers, the gasification agent enters the reactor at a lower point, this changes the position of the different thermochemical conversion zones. While drying still takes place at the top, it happens in an anaerobic atmosphere. When compared to the up-draft gasifiers, the product gas exists the reactor at considerably higher temperatures, but with lower tar generation. The main advantage is the high product gas quality, this comes at the cost of high fuel quality requirements, including fuel particle size and water content. Furthermore even temperature distribution is crucial to ensure complete splitting of long carbon molecule chains which form in the upper part of the gasifier.[58]

2.6. Use of additives

As stated before, the main difficulty when using pellets made from non-woody biomass is the softening and subsequent melting of ash, which potentially clogs reactors. To counter those negative effects, additives may be added to the biomass feed. These are minerals or chemical compounds, which increase the ash softening temperature by interacting with the feed material in different ways. Lessig[6] and Wang et al.[25] summarised the four main interactions:

- Chemical reactions of additives and compounds, specifically through so called alkali-getter effects, can lead to the formation of new compounds with higher SST/DT out of the existing low SST/DT materials. This also has the added advantage of removing problematic compounds, in particular potassium, from the exhaust. Overall, chemical reactions are preferred to other methods, as they are best suited to prevent the leakage of KCl by binding it. Depending on the elemental composition of the the additive this has different specific effects. Regarding their chemical composition, additives can be classified into four categories, aluminium silicate based additives: sulfur based additives, calcium based additives and phosphorous based additives:
    - Aluminium silicate based additives form potassium-aluminium-silicates by taking the present potassium out of existing chlorine-potassium-compounds and further bind the potassium within the ash. Examples for such additives are kaolin, bentonite, coal fly ash, emathlit, zeolithe and sludge.
    - Calcium based additives form calcium-silicates with higher ash softening temperatures out of potassium-silicates, with the potassium converting into gaseous phase. Examples for such additives are dolomite and lime.
    - Sulfur based additives convert KCl into K_2SO_4, this increases the SST from approx. 770 °C to approx. 840 °C. Typical examples for such additives are ammonia- and aluminium-sulfate.
    - Phosphorous based additives form potassium-phosphates out of potassium compounds, which can in turn further react with any present calcium. Through this reaction chain, fewer calcium-silicates, which have lower SST/DT, are formed. Typical examples are ammonia phosphate and phosphoric acid.
- Physical adsorption captures aerosols and fines within the porous surface of the additive material. These loaded particles subsequently leave the reactor and prevent further interaction with both fuel and product materials.
- Addition of inert materials with high melting temperatures like SiO_2 or Al_2O_3 increase the overall ash SST/DT.
- Dilution decreases the risk of agglomeration which further helps to limit ash melting.

Lessig[6] analysed five different additives reviewing their viability when used with non-woody biomass, specifically hay, straw, corn cobs and miscanthus. The additives in question are kaolin, bentonite, coal fly ash, dolomite and lime, the analysis was done using fuel index calculations described in chapter 2.2.4. Indices above 3 mean ash SST below 1000 °C. In order to reach ash SST beyond 1100 °C an index value between 2 or 3 is required, for SST above 1200 °C values below 1 are necessary. The index used is the aluminium-index, which is shown in 14. This index is used since it takes all relevant elements into consideration.

$$\frac{\mathrm{Si}+\mathrm{P}+\mathrm{K}}{\mathrm{Ca}+\mathrm{Mg}+\mathrm{Al}} \text{in mol/mol} \tag{14}$$

A graphic summary of the results calculated by[6] is displayed in table 6. When analysing this data, it can be concluded that: The general addition of additives always lowers the fuel index value and subsequently increases SST. Additionally, an increase of additive amount also leads to further decrease of index value and increase in SST. Within the table, red indicates not usable materials with ash SST <1000 °C, orange indicates SST around 1100 °C, yellow indicates SST approaching 1200 °C, green indicates SST >1200 °C and grey indicates no available index-data. To asses these calculations, green is expected to be usable, yellow has a high chance to be usable, orange is likely to not be usable and red is expected to not be usable.

Data regarding the fuels suggests promising results when using hay, as more than half of the analysed additives should yield acceptable results, while when using straw, corn cobs and miscanthus, only some specific additive types and amounts yield favorable results.

Looking at the additive types and amount directly, both dolomite and lime suggest favorable results, especially when added in higher quantities, while kaolin may also be a viable alternative when used at at least 5 % of total pellet raw mass. Both bentonite and coal fly ash are very unlikely to yield favorable results.[6]

Table 6.: *Comparison of the viability o different additive types when used with specific biomass types.[6]*

Additive in m%	Fuel Hay	 Straw	 Corn cobs	 Miscanthus
No additive	2,49	5,80	5,76	5,98
2,5% Kaolin	2,08	3,65	3,77	3,79
5% Kaolin	1,85	2,84	2,95	2,95
2,5% Bentonit	2,38	4,44	4,51	4,58
5% Bentonit	2,30	3,79	3,88	3,91
2,5% Coal fy ash	2,19	3,95	4,05	4,10
5% Coal fly ash	2,01	3,18	3,29	3,29
2,5% Dolomite	1,63	2,73	2,87	2,86
5% Dolomite	1,22	1,80	2,21	1,89
7,5% Dolomite	<1	n.a.	n.a.	n.a.
2,5% Lime	1,66	2,82	2,97	2,96
5% Lime	1,25	1,87	2,00	1,97
7,5% Lime	1	n.a.	n.a.	n.a.

When further comparing the previously mentioned observations to the interaction types described by [25], further possible reasons for the favorable results for kaolin, dolomite and lime, as well as the non-favorable results for bentonite and coal fly ash can be found. As graphically shown in table 7, bentonite and coal fly ash have only one way of interaction with the biomass feed material, while kaolin and dolomite may interact in two ways and lime in all four ways.[25]

Table 7.: *Interaction type of different additives[25]*

Interactions	Additives Kaolin	 Bentonite	 Coal fly ash	 Dolomite	 Lime
Chemical adsorption	Yes	Yes	Yes	Yes	Yes
Physical adsorption	Yes	No	No	Yes	Yes
Inert materials	No	No	No	No	Yes
Dilution	No	No	No	No	Yes

2.7. Economic assessment

Lessig[6] conduced an economic assignment, with the results being displayed in table 8. It has to be noted that this table does not include transportation costs and taxation. The transport cost is particularly relevant for samples with additives, since the additives have to be transported to the factory separately. Based on this assessment, wood chips still remain the cheapest fuel type, the pellets made from non-woody biomass however outclass the pricing of wood pellets, even with the use of additives. Due to market volatility, this may vary over time. Hereby, the waste products of straw and corn cobs are notably cheaper than the purpose-grown hay and miscanthus. It can also be said that the location of the production plant significantly influences the viability of different fuel types, as transport costs and raw material cost can locally vary. This also goes hand in hand with the regional assessment done by [5]. To summarise, pellets made from non-woody biomass with additives can be an alternative to wood-based fuels, it is however crucial to take market volatility and local availability into consideration.[6]

Table 8.: *Overview of expected cost[6]*

	Cost in €/t					
Additive	Hay	Straw	Corn cobs	Miscanthus	Wood pellets	Wood chips
No additive	115	82.5	80	110.0	195	90
5 wt% Kaolin	153.4	123.9	115.2	149.4	n.c.	n.c.
5 wt% Dolomite	137.1	106.6	97.9	132.1	n.c.	n.c.
5 wt% Lime	138	107.6	98.9	133	n.c.	n.c.

2.8. Political and public debate

The European fit for 55 package is a major legislative directive with the aim of achieving a 55 % reduction in greenhouse gas emissions until the year 2030. On specification of this package is the REDII[61] guideline which defines several actions and thresholds to achieve the set goals. In 2021, a revision process for the REDII[61] guideline was started, which resulted in the creation of the REDIII[62] guideline. This revision sparked a heated debate, especially regarding the proposed changes to the use of forest biomass. The proposed changes regarding biomass are the phasing out of electric production from biomass from 2026 onward, as well as preventing several raw materials to be used for energy production, while minimisation the risk of market distortion and use of renewable energy systems (RES) schemes.[62]

Article 29[62] specifies the changes further. The preservation of primary and highly divers forests, as well as peatlands is a major goal. To achieve this, forest biomass should also be partly excluded from energetic use, similar to some agricultural biomass, in particular food. Furthermore, the sourcing of any biomass for energy production which originates from primary forests should be prohibited. Primary forests are defined by[63] as "forests with no significant disruption of ecological processes through human activities, which also show natural regeneration of native species". National financial incentives to use saw/veneer logs, stumps or roots for purely energetic use should be banned. In addition the threshold for the application of the EU sustainability criteria, as well as greenhouse gas emission thresholds is lowered to installations $\geq 5\,\mathrm{MW}$ + total rated thermal capacity and is applicable to already existing installations.[62]

Several organisations have made advisory and critical comments regarding the REDIII[62] guideline. The European Committee of the Regions (CoR) states that the sustainable production of biomass is crucial for the preservation of biodiversity and general environmental protection. It also notes however, that the introduction of new and more strict thresholds and limitations for all existing small installations poses a threat to the stability of the legal framework.
Fern, a self declared movement for the protection of forests in the EU and UK, criticised the REDIII[62] guideline for still supporting the general use of biomass and states that additional forest protection is necessary, otherwise coal fired power plants could be incentivised to switch to biomass as fuel.[62]

Bioenergy Europe, alongside several other organisations, released a joint statement criticising the REDIII[62] guideline. According to them, the forced cascading use of biomass could lead to a distortion of the raw material market, which would contradict aim of the REDIII[62] guideline to avoid such issues. Furthermore they criticize an improper involvement of the affected member states regarding the formation of forest no-go areas, as such areas could lead to difficult long-term planning and possible conflicts with property rights of affected nations, states or individuals. This could be amplified by continuous short therm revisions, which may hamper planning even further. As a final criticism, Bioenergy Europe stated that retroactive measures may cause a substantial socio-economic impact since those measures would hit small installations more than larger ones, due to their smaller administrative capacity and smaller size of installations. [64]

The Landwirtschaftskammer Österreichs (Austrian agrarian chamber) also voiced critical comments towards the REDIII[62] guideline. Firstly, the chamber criticized the general aim of the fit for 55 package, as the long legislative process makes implementation of the various measures not feasible. Secondly, the effects of expensive infringement processes could severely hurt affected nations, states and companies, which become more likely due to the aforementioned long legislative processes. Similar to Bioenergy Europe, the Landwirtschaftskammer Österreichs also criticizes a lack of coordination with the most affected countries, namely Sweden, Finland, Lithuania, Estonia and Austria, as well as the reduction of the application output to $\geq 5\,\mathrm{MW}$ + total rated thermal capacity and the short term reevaluation. They also specifically mention that the introduction of mandatory cascading for biomass, but not for fossil fuels seems counterproductive to the wider goals. As an alternative solution the

Landwirtschaftskammer suggests using the OECDs "Risk-Based approach" instead of no-go areas for forest biomass due to its clearer definitions and limitations. As a final point of critique, the Landwirtschaftskammer considers the definitions for wood quality suitable for energetic use inadequate, since they do not consider regional characteristics and availability.[65]

To address the criticism, but also to further combat the effects of climate change, the REDIII[62] guideline was revised in 2023. Wien Energie [66] provides as summary of the revised REDIII[62] guideline[67]. The most important points are: The percentage of renewable energy sources of all member states, should be increased to 42 % overall, and 49 % in the building sector. Additionally, the approval procedures for new renewable energy projects should be capped at 12 months to considerably speed up the process. The EU has also given in to the demands of the industry, wood will remain as a renewable energy source, even when used for energy production. Some limitations are however put in place, as member states should not provide financial support for production of energy from saw logs, veneer logs, industrial grade roundwood, stumps and roots. Furthermore, wood products should be used according to their highest economic and environmental added value, which includes the extension of service live, re-use and recycling.[66][67]

3. Experimental part

3.1. Methodology

From a practical point of view, the methodology of the thesis can be categorised into 4 distinct steps: The preparation of the fuel, which means using a hammermill to create a suitable particle size, the pelletisation of the prepared fuel, the incineration and subsequent creation of ash, and the analysis of the ash through use of an ash-microscope to determine the different ash softening and melting points.

3.1.1. Fuel preparation

Hay, straw and miscanthus are all delivered in fibrous form, with straw and hay being long, fibrous grasses, while miscanthus is already pre-cut to shorter fibres, it is however still too large for pelletisation. The corn cobs on the other hand are already delivered in granulated form, thus no further preparation is required. The unprocessed fuel, as delivered, is shown in fig.13, while the used additives are shown in fig.14.

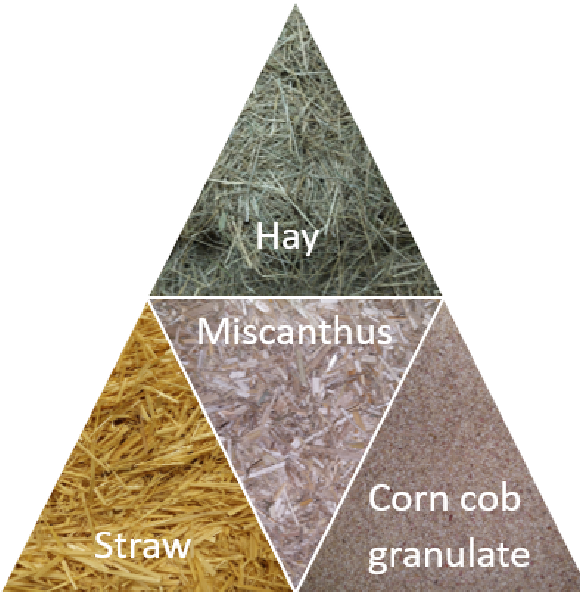


Fig. 13.: *Raw fuel before cutting*

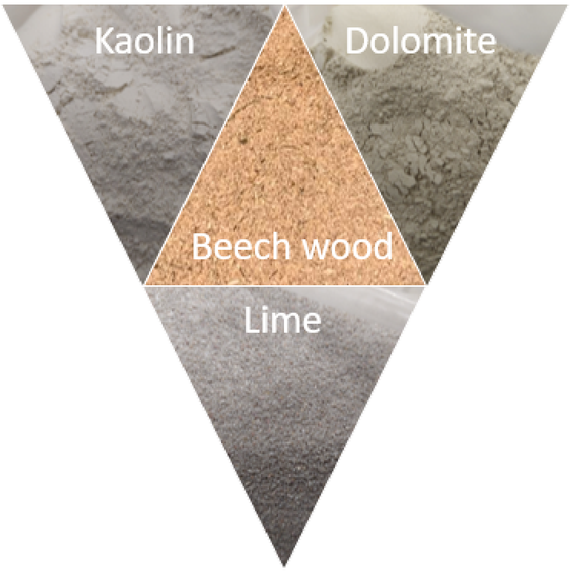


Fig. 14.: *Used additives*

The milling of the fuel is done using a Fritsch Pulverisette 19 cuttingmill. Fig. 15 and fig. 16 show the used cutting mill in closed and open configuration. Two sieving sizes are available, 2 mm and 4-5 mm.Trials are conducted with both sieves, based on which the 4-5 mm is chosen due to lover dust formation and slightly better pelletisation. The sieves are shown in fig. 17. For the milling process, raw material is inserted at the top of the device and pushed down using a plunger. To avoid clogging, material should not be pushed into the mill too fast, especially when using harder materials such as wood. Fig. 18 shows the knife which is used to cut the material.

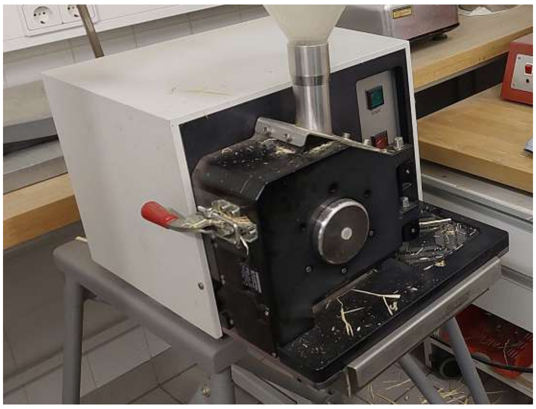


Fig. 15.: *Closed cutting mill*

Fig. 16.: *Open cutting mill*

Fig. 17.: *2mm sieve and 4-5 mm sieve*

Fig. 18.: *Rotating knife within the cutting mill*

3.1.2. Pelletisation

Pelletisation is done using a Cissionius PP200 7.5 kW pellet press, shown in fig.19, with the technical data presented in fig.20. For the press, several matrix diameters are available, in this thesis only the 8 mm matrix is used. According to the technical data given by the manufacturer, the capacity of the device is 100-150 kg/h for wood and 100-250 kg/h for livestock-fodder, whereas hay, straw, miscanthus corn cob granulate can be classified in the second category. Due to the experimental nature of the trials, only batch-operation is practised, with each batch being ≤ 2 kg.

Fig. 19.: *Pellet press*

CISSONIUS

Cissonius GmbH Liebenwalder Chaussee 19 16792 Zehdenick Germany

Model	PP-200	Weight(KGS)	230
Power(KW)	7.5	Packing(CM)	113*48*92
Output(Kg/Hr)	100-150	Manufacture date	201

www.pellet-mill.de CE

Fig. 20.: *Data of the pellet press*

For the pelletisation, the already cut material is mixed with additive and water, in general 20 wt% water is added at the beginning, if strong heating is observed, additional water may be added to avoid clogging. The pellet press is started and material is added as soon as the machine finishes its up-shift. During the process fuel is continuously added by hand until each batch is finished. In general, 2 passages of the material are done, with more passages only being relevant for corn-mixtures. For several mixtures is possible to do several different combinations in succession without stopping the press, this speeds up the process significantly but poses a risk of cross-contamination. In case of clogging, the press has to be opened and the matrix unblocked using a drill bench.

For corn cob pellets, 6 passages are conducted due to higher feed material hardness, for all other feed materials, 2 passages are done.

The storage of pellets proves to be an additional challenge. With considerable baseline moisture content, as well as the added water, the final pellets have a considerable wetness, even after the drying which occurred during pelletisation. Because of this, mold becomes a risk. The pellets produced in the trial runs were left in sealed plastic containers for about 3 weeks, after which mold was detected in the hay, corn and miscanthus samples, no visible mold was however detected in the straw samples. Because of this, all further samples are dried after production.

After the pelleting, the product pellets are sieved manually through a 4 mm sieve, to separate small particles and abrasions. The finished pellets are then dried for at least 24 h on a dry towel in a room with sufficient draft, to prevent the formation of mold, rot or fouling.

3.1.3. Incineration

Further analysis is done using ash, therefore the previously produced pellets must be incinerated. This is done using a muffle furnace. According to the DIN EN ISO 21404[7] standardisation, the incineration program is as follows: In the first stage, the sample is heated up to 250 °C for 30 min, it is then held at this temperature for one hour. In the second stage the sample is again heated up for 30 min up to 550 °C and held at that temperature for at least 2 hours, although more time is required in case of larger sample sizes. To ensure sufficient incineration, the second sage is held at 550 °C for 480 min or 8 h, as shown in fig. 21. If the produced ash is not used right away, and additional after-glowing of 1-2 hours may be conducted to eliminate any accumulated moisture content.

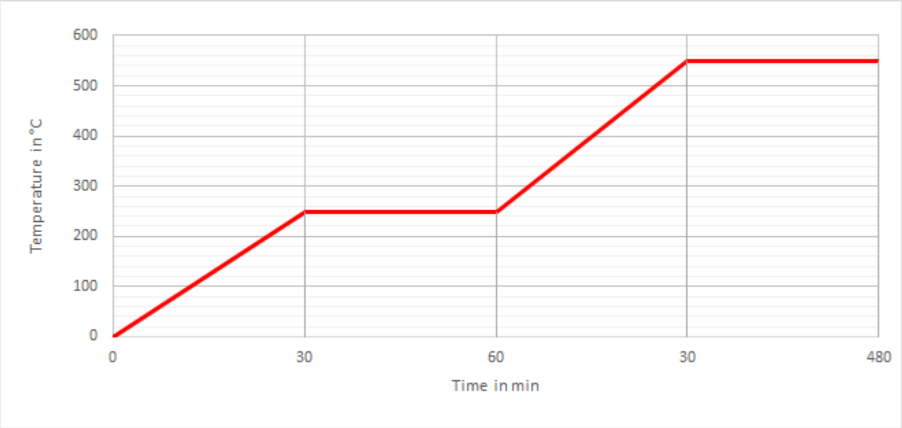


Fig. 21.: *Temperature program used for pellet incineration*

The muffle furnace used has a capacity of five samples, which are placed into ceramic bowls. The pellets are slightly shifted towards the edges of the bowls to maximise surface area. For each sample 150 g of pellets are incinerated, the only exception being sample F, were only 113 g of pellets are available. After incineration, the ash skeletons are transferred into sample containers and stored until used.

3.1.4. Ash microscope analysis

Before the analysis, a standardised sample cylinder must be produced. To analyse a sample a sufficient amount of ash is put into a mortar, finely ground and scooped into a small mound. Several drops of ethanol are added to the mound, afterwards a spatula is used to scoop up a sufficient amount of ash. The ash is placed into the largest hole within the mould. In the next step, the ash is pressed into a cylindrical form using the hand press, this is done multiple times to ensure the created sample cylinder is of suitable size. The mould is then opened and a smaller press is used to place the cylinder on a sample plate. Fig. 22 and 23 show the mould used to create sample cylinders. For all samples, the top left hole, shown in fig 22 is used.

Fig. 22.: *Closed mould*

Fig. 23.: *Open mould*

The ash microscope itself consists of multiple parts, as shown in fig.24. These are, from left to right: The heating element, the lamp, the water cooled sample chamber, the camera and, not in the picture, a computer containing the analysis-software. Fig. 24 also includes a close up of the heating chamber during analysis, sitting at >1000 °C. Before starting the analysis, water cooling, heating element and lamp must be turned on. The sample is placed upon a flattened spot on the cylindrical heating element and slid into the measurements chamber, which is subsequently locked. The camera can be adjusted to centralise on the position of the sample. This needs to be done before each experiment, since the central sample hold tends to slightly twist during the heating process. Once the process is started, the sample is heated up with a rate of 10 °C per minute up until either 1500 °C or the respective flow temperature are reached. Results are automatically collected by the software, which presents recommended temperature points for the SST, DT, HT and FT.

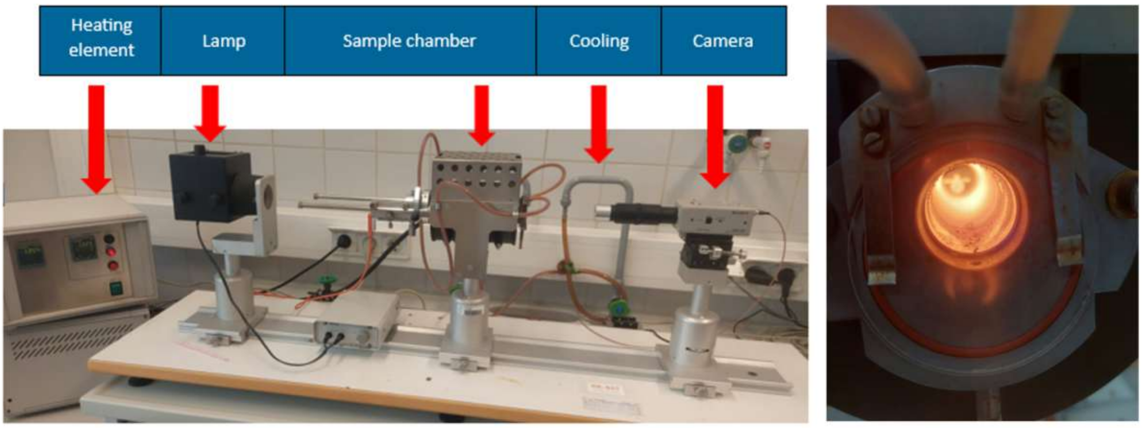


Fig. 24.: *Ash microscope with close up of the sample chamber during an experiment*

3.1.5. Tested samples

An overview of all tested samples is given in table 9. At total of 20 samples are tested.

Table 9.: *Overview of all tested samples*

	Sample			
Additive	Hay	Straw	Corn cobs	Miscanthus
No additive	A	F	K	P
5 wt% Kaolin	B	n.c.	L	n.c.
5 wt% Dolomite	n.c.	G	n.c.	Q
7.5wt% Lime	C	H	M	R
33 wt% beech wood	D	I	N	S
66 wt% beech wood	E	J	O	T

3.2. Trial runs

3.2.1. Background

At first, trial runs with various amounts of additional water added to the feed material are conduced. These trial runs are done using only the fuel material itself, with no additives admixed. When adding no water, pellet formation does not take place, instead the feed material falls directly through the matrix. According to scientific data, as described by Garcia et al. [43], ideal moisture contents range from 6 wt% up to 35 wt%, depending on the feed material, with around 16 wt% being the ideal value. Trial runs in this thesis have shown the best results when adding 20 wt% of feed material weight in water to the mix.

For work safety several precautions should be taken: Due to noise, heat and dust loading, personal protection equipment (PPE) is required. The milling of wood hereby creates notably more noise than the non-woody raw materials used. Harder materials such as granulated corn cobs or wood increase the risk for such clogging. Additionally, the use of harder materials leads to more heat generation. Due to the low density of some the base material, the cutting process can be very time consuming, therefore automation is highly recommended for industrial uses. Hay and straw are particularly notable in this regard. Regarding work conditions, dust loading occurs with all tested fuels, however miscanthus appears to be particularly prone to it. To mitigate the effects, a makeshift extraction using a vacuum cleaner is utilized. In addition the frontal holding screws of the lock, which hold the front lid, as shown in fig 15 in place, may loosen up due to vibrations during operation.

The main issue during batch-pelletisation is the tendency of clogging, which is particularly noticeable when producing mixed material pellets containing wood. In case of clogging, the press has to be opened and the matrix subsequently drilled open using a drill bench with a 7.5 mm to avoid damaging the matrix. The level of clogging varies between the different holes within the matrix, those closer to the edge and center clog less than the middle ring. It is therefore assumed that fuel compression is weaker at he edge and center of the matrix.

When unblocking the matrix, the clogged pellets are only partially destroyed by the drill, thus most of the stuck pellets can be retrieved and used. The appearance of such retrieved pellets differentiates quite notable from pellets directly coming out of the press. In general they have a noticeably darker, brownish mantle and higher hardness. It is assumed that the longer dwell time within the hot matrix leads to a sort of "after-baking" with slight charring of the surface area.

Different notable pelletisation properties, depending on the fuel are: The pelletisation of both hay and straw functions best, with the least heat generation, dust loading and clogging tendencies. Miscanthus leads to the highest dust loading. The high hardness of corn cob granulate leads to more heat buildup, for their respective pelletisation higher water content and multiple passages (up to 6) lead to better results. Production of mixed wood/non-wood pellets is comparatively more difficult due to very noticeable heat generation and high tendency to clog. Furthermore, feed material with higher hardness, i.e. wood and corn cob granulate facilitate even more noise generation and increases the chance for fuse failure due to overload. For each used non-woody biomass type, trial runs were conducted to assess the specific requirements for the pellet production.

3.2.2. Straw trial run

At first, a trial run with straw as feed material is conducted in the following order:

1. Barley straw at a size of 2 mm with no added water is pelleted
2. No pelletisation takes place
3. Then, three litres of barley straw with 250 mL of water added are pelleted
4. Crumbly pellets form
5. An additional 250 mL of water is added
6. Darker, more stable pellets form, however the matrix clogs after a short time

It is assumed that the high water addition leads to the wet slurry entering the holes in the matrix, drying and hardening there, while it is not possible to exert sufficient pressure through the still wet slurry on top. This leads to the wet slurry being milled by the rolling mills while the material in the holes is continuously dried and hardened. The pellets produced in the first trial run are shown in fig 25 and fig 26.

Fig. 25.: *Barley straw pellets after one passage*

Fig. 26.: *Barley stray pellets after two passages*

3.2.3. Hay trial run

The second trial run is done using hay as feed material:

1. 2.5 litre of hay at a size 4-5 mm, with 250 mL water added are pelleted
2. 3 passages through the pelleting press are done
3. Formation of wet, crumbly pellets
4. Three litres of hay at 2 mm, with 225 mL of water added are pelleted
5. 4 passages through the pelleting press
6. Formation of crumbly pellets

The produced pellets are shown in fig. 27. Several observations can be made. First of all, a continuously lighter colour, depending on the number of passages is notable, this can be attributed to a decrease in moisture content. Secondly, the presence of visible fibers also decreases with further passages, which is caused by the pelleting process also functioning as mill.

Fig. 27.: *Hay trial runs, 4-5 mm, 1-3 passages (upper picture); 2 mm, 1-4 passages (lower picture)*

3.2.4. Miscanthus trial run

The third test is done using miscanthus

1. 2.5 litre of miscanthus at 2 mm, with 125 mL water added
2. Significant dust loading during operation takes place
3. Agglomeration around pockets of moisture leads to the formation of pellets
4. Timely clogging occurs, addition of water during pelleting does not resolve the issue
5. Soaking the matrix leads to no improvement, declogging is only possible through drilling possible
6. Serviceable pellets form

The produced pellets are shown in fig.28 and fig.29. They exhibit a homogeneous structure and a somewhat hard mantle. The pellet ends also show inflated ends, furthermore the aforementioned dust loading can also be seen in fig.28.

Fig. 28.: *Trial run of miscanthus pellets*

Fig. 29.: *Close up on miscanthus pellets*

3.2.5. Corn cob trial run

1. Corn cobs are already bought in granulated form
2. Pelleting of pure granulate without the addition of water forms no pellets
3. 500 mL of corn cobs, with 100 mL water added
4. Additional water added during the operation
5. Pellet press also functions as mill, some granulate is milled to powder
6. Formation of serviceable, but short and heterogeneous pellets, with some granulate remaining as part of the pellets.
7. Firstly, only small pellets form, up to 6 passages necessary to create serviceable pellets

It can be concluded from the corn cob pellet trial runs, that due to the high hardness and low innate wetness, more pass troughs are required. Additionally the mentioned milling through the pelleting process also creates dust, which can be seen in fig.30. The resulting pellets after 6 passages, as displayed in fig.31 show a smooth mantle with darker spots. Small cracks are also visible, those are most likely formed near granulate particles which retained their original form and were not milled during pelleting.

Fig. 30.: *Trial run of corn cob pellets*

Fig. 31.: *Close up on corn cob pellets*

3.2.6. Summary of trial runs

A short summary of the results from the trial runs is shown in table10. Regarding the moisture, 20 wt% added water as a starting point is chosen, depending on the heat generation during pelleting and amount of passages, additional water may be added while operating. For the feed material, 2 mm is chosen due to the easier cutting of the material and comparable results. Homogeneity, structural integrity and pellet hardness are all connected due to the pellet press also functioning as a mill. A higher degree of milling leads to more homogeneous pellets, which in turn are harder and exhibit better structural integrity. This is achieved by passing the material through the press more often, but it continuously dries the material which increases the risk of clogging. Ultimately, 2 passages yield the most favorable results. Corn hereby remains the exception, since the high hardness of the granulate makes multiple passages necessary to create homogeneity.

Table 10.: *Summary of the trial runs*

Property	Conducted tests	Preferred result
Moisture	5 wt%, 10 wt%, 20 wt%	20 wt%
Feed material size	2 mm and 4-5 mm	2 mm
Homogenity	Variable passages	Corn: 6 passages, Rest: 2 passages
Hardness	Variable passages	Corn: 6 passages, Rest: 2 passages
Structural integrity	Variable passages	Corn: 6 passages, Rest: 2 passages

4. Results and Discussion

4.1. Pellet Production

The following characteristics of the pelleting process are determined experimentally: Considerable amounts of feed material are lost during the process, with total mass losses ranging from 3 % up to 75 % of the initial mass, depending on the material combination. Data hereby suggest ascending losses when going from hay to straw, corn cobs up miscanthus with the highest mass loss percentage. Additionally, using wood as additive also leads to higher losses. Potential causes for this are: Both wood and corn cobs have a notably higher hardness compared to the other used fuel materials, which in turn leads to an increased risk of matrix-clogging. When cleaning the matrix by drilling it open, only some of the pellets may be reclaimed, thus material losses occur. Miscanthus on the other hand creates the most dust loading, it is therefore assumed that parts of the material are discharged during pelleting. It has to be noted though, that the manual batch operation of the pelleting means the collected data may has some variance, further testing is required for more accurate determination of the process efficiency.

On of the thesis main objectives is to determine if the production of serviceable pellets from non-woody biomass with and without additives is possible. The experiments have hereby shown that this is indeed possible for all tested combinations, although the actual quality of these pellets varies. The results are shown in fig 32 to fig. 35.

When looking at the hay samples, shown in fig. 32, the following observations are made: All hay pellets exhibit an inhomogeneous structure, with visible fibers remaining. The pellet colour is brown, with the pure hay pellets in sample A being the lightest tone, samples B, C and D being slightly darker and sample E the darkest in comparison. Furthermore, sample A is the softest, samples B and C slightly harder and both wood-containing samples, D and E displaying medium hardness. Sample E also has a smooth mantle surface.

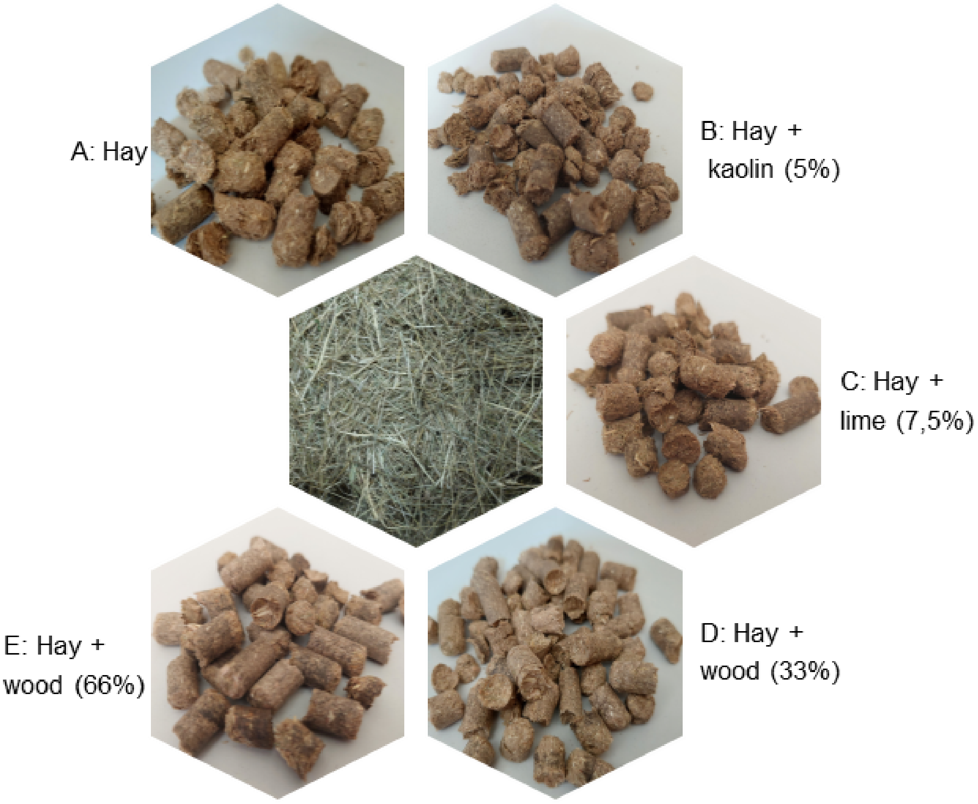


Fig. 32.: *Visual summary of all hay-pellet samples*

For the straw based pellets, shown in fig. 33, the following observations are made: The general colour is a light beige, the addition of wood leads to darkening in sample J, although no darkening is visible in sample I. In general, the samples show an inhomogenous structure, although less so than hay, since fibers are not directly visible. The overall hardness is higher than that of hay pellets, but not as high as some of the other fuel samples. In sample I, some pellets are less compressed, showing a more inhomogenous structure. Sample J shows a slightly dark mantle.

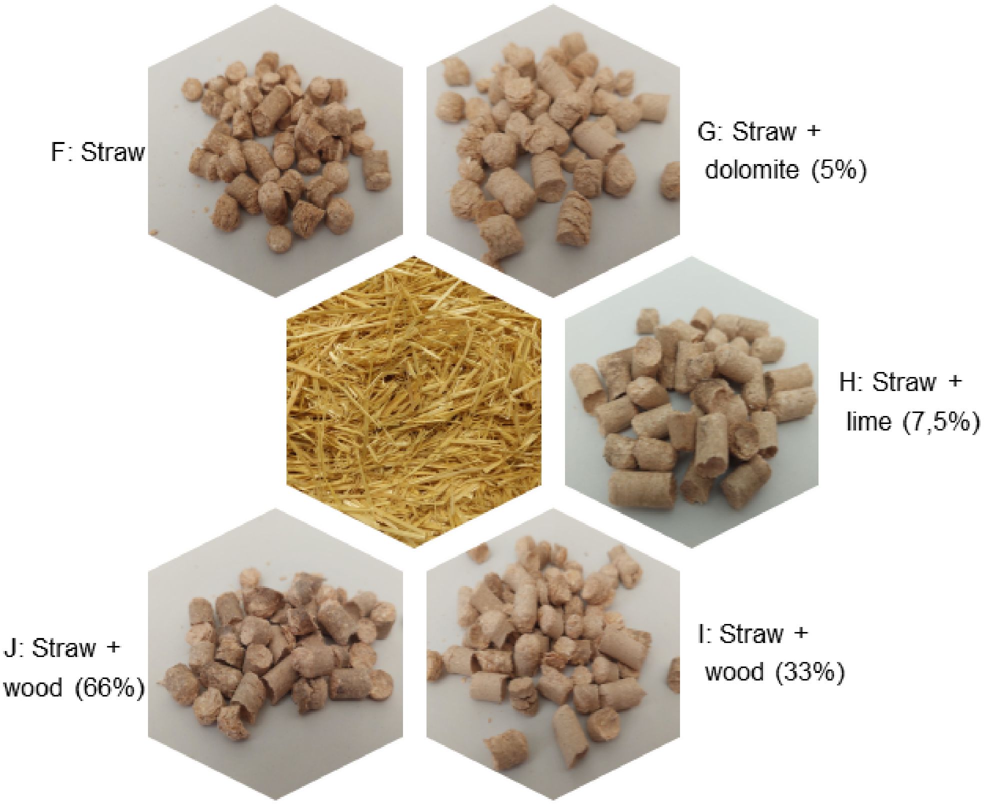


Fig. 33.: *Visual summary of all straw-pellet samples*

The visual data for corn cob pellets, displayed in fig. 34 suggests that homogeneity depends on the degree of milling of the feed material during the pelleting process. In samples K and L the presents of non-ground granulate is particularly noticeable, this also leads to the presents of fault lines on which the pellets show an increased tendency to break, which in turn leads to more crumbly pellets. Those fault lines can however be also seen in sample M, which has a more homogeneous structure than K and L. The addition of wood facilitates a darker mantle, as can be seen in samples N and O, although this does not occur equally on all pellets. Generally speaking, if pellets are sourced from the cleaning/drilling of the matrix, they have a higher tendency to form a dark mantle.

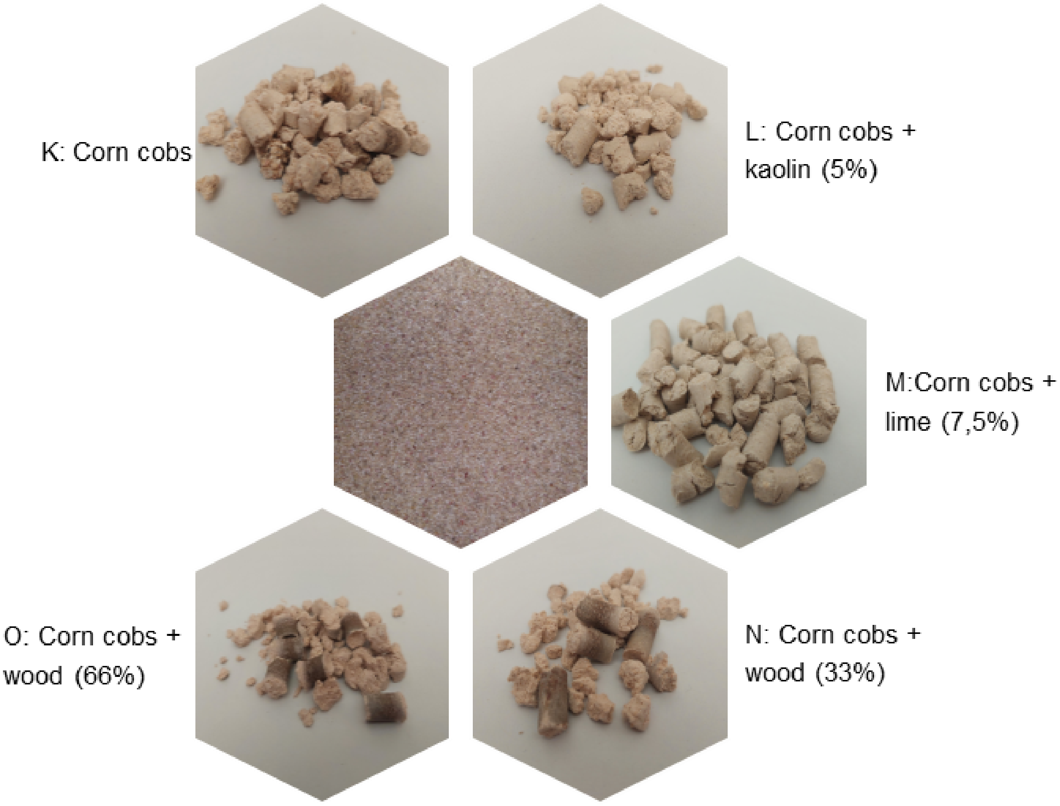


Fig. 34.: *Visual summary of all corn-pellet samples*

The miscanthus pellets, shown in fig.35, similar to the hay pellets, display an inhomogenous structure with visible fibers in all samples, although less clearly in sample S , which is also softer than the other samples and also has break lines as observed in other fuel samples. Samples P, Q, R and T are all rather hard with a smooth mantle. Sample T is the only miscanthus sample which has a dark mantle.

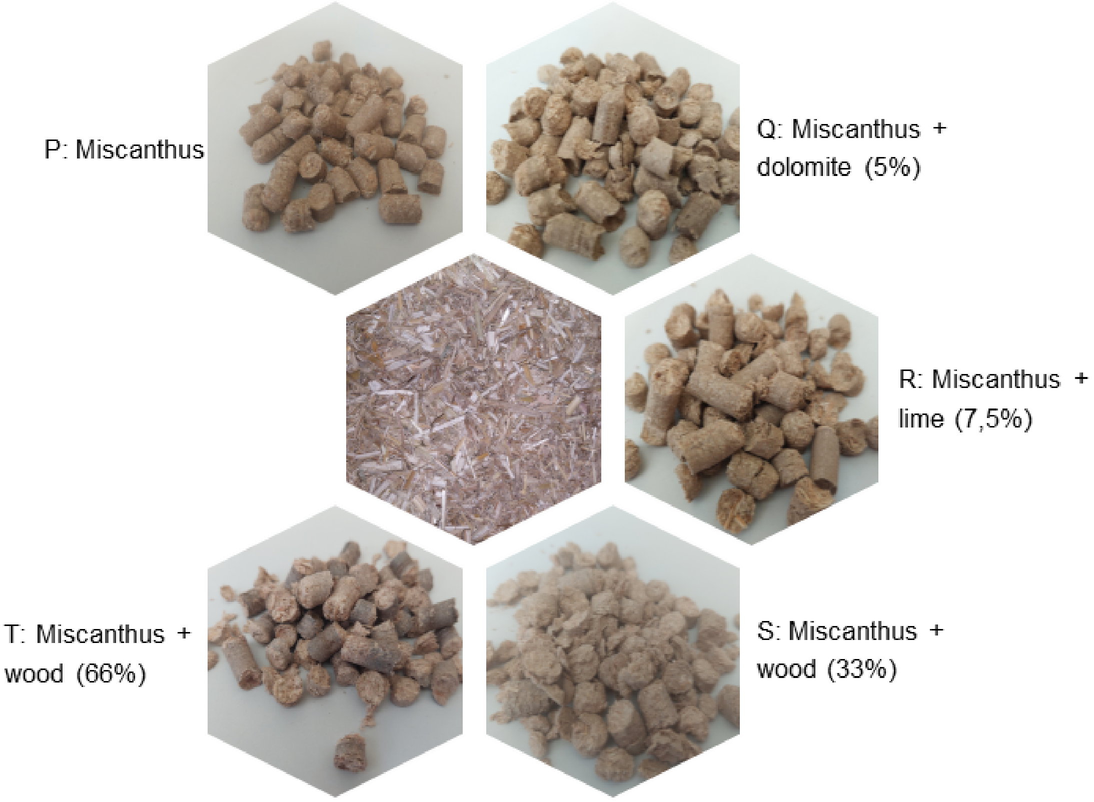


Fig. 35.: *Visual summary of all miscanthus-pellet samples*

4.2. Incineration

The ash skeletons of the hay samples, as visualized in fig. 36, all have a grey texture with orange colouring at the mantle. In sample B, the addition of kaolin leads to a darker, more reddish shade of orange, while he addition of lime in sample C leads to a lighter, yellowish orange tone. Overall, the different hay samples show comparable visual properties though.

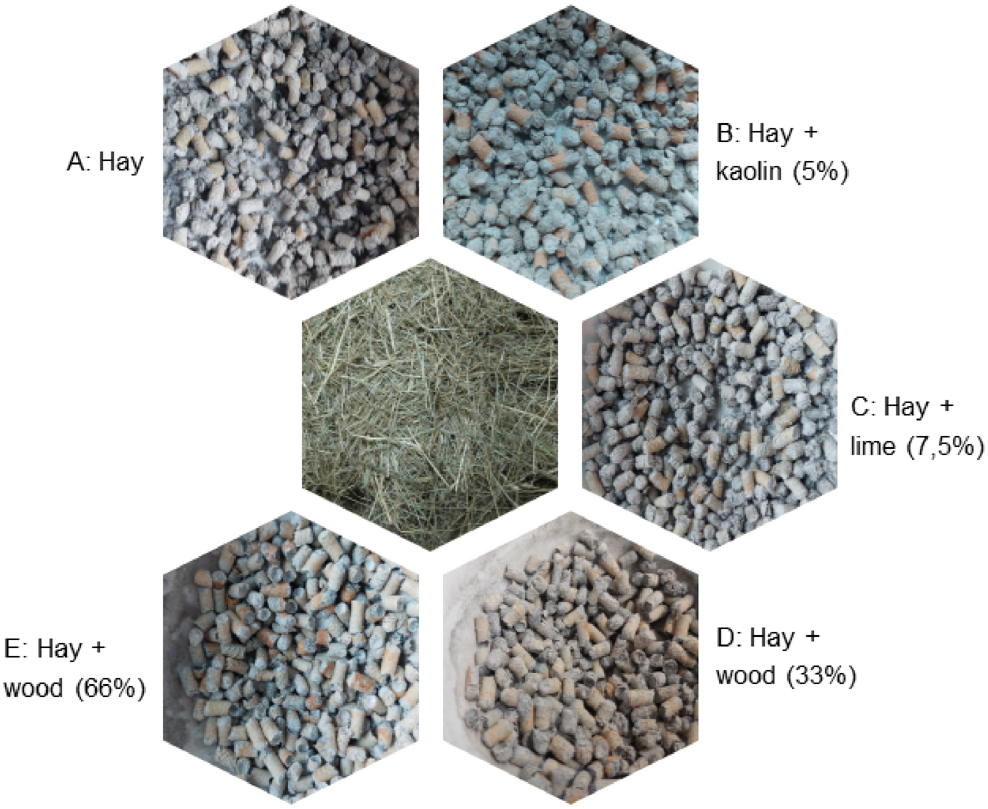


Fig. 36.: *Visual summary of all hay ash skeletons*

Similar to the hay samples, the ash skeletons of straw samples, depicted in fig 37 display a grey base colour with an orange mantle. The orange colour is more pronounced in sample F consisting of pure straw, while the addition of dolomite and lime leads to less pronounced orange. The wood/straw mixed pellets are more crumbly when compared to other samples.

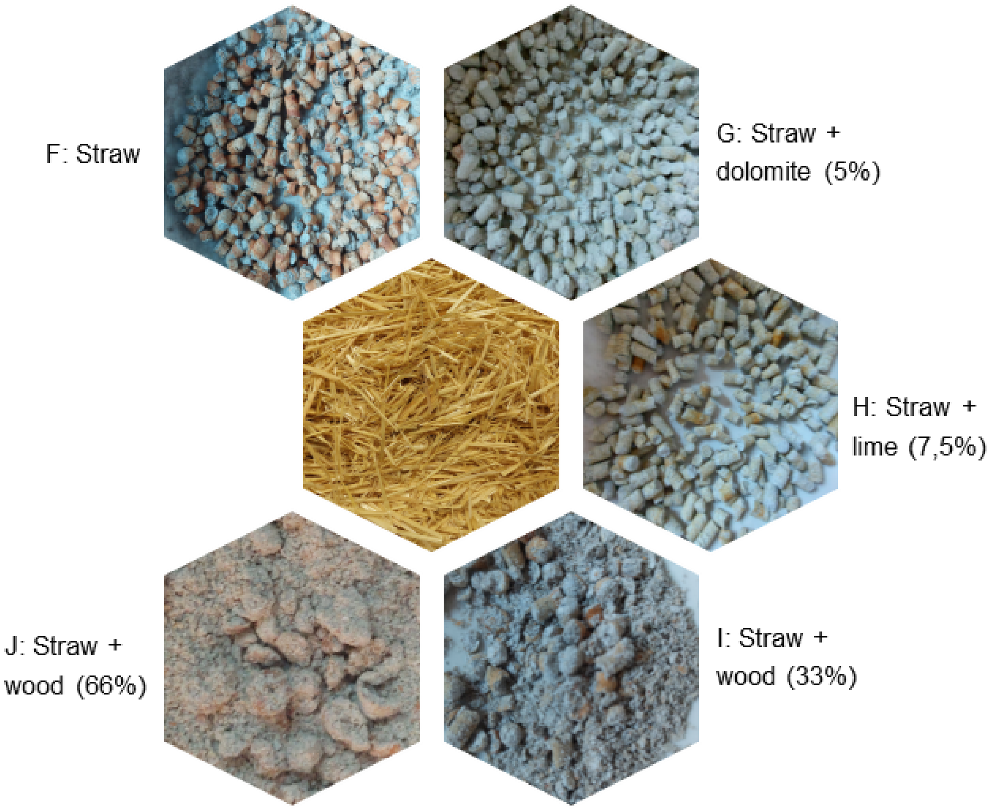


Fig. 37.: *Visual summary of all straw ash skeletons*

The ash skeletons of the corn cob samples are shown in fig. 38. The base colour of the ash is beige, with the different additives creating notable variations in colouring. Hereby, kaolin leads to crimson coloured ash, while lime turns the ash almost white. Both samples L and M have a homogeneous colouring, while pure corn, as well as the pellets with added wood lead to a notable presence of orange around the mantle. For pure corn pellets, the total amount of ash is considerably lower than in blended samples and sample with additives.

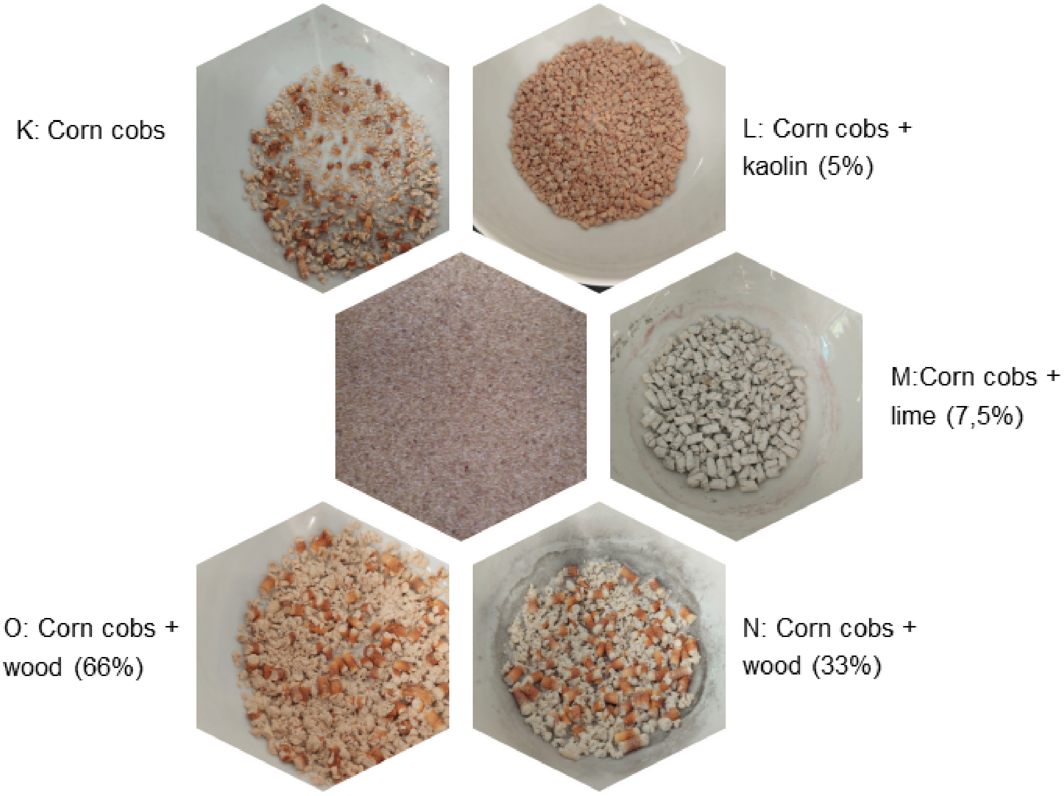
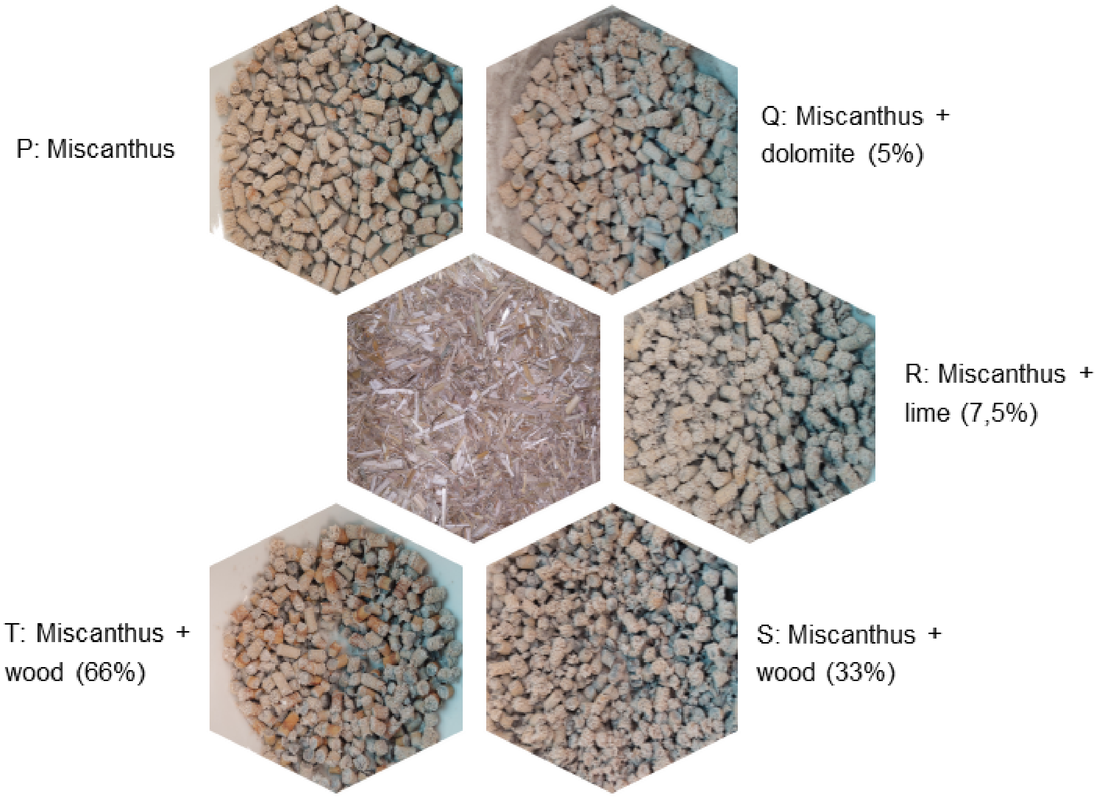


Fig. 38.: *Visual summary of all corn ash skeletons*

Visually, the miscanthus pellet ash displays a grey/brownish base colour, with a light orange mantle, as depicted in fig. 39. Sample T is he only one with a more pronounced orange mantle, other additions do not lead to notable deviations of the base colour. All miscanthus ash samples exhibit a coarse outer structure, which is more notable than with other fuel types.


P: Miscanthus

Q: Miscanthus + dolomite (5%)

R: Miscanthus + lime (7,5%)

T: Miscanthus + wood (66%)

S: Miscanthus + wood (33%)

Fig. 39.: *Visual summary of all miscanthus ash skeletons*

To summarise, the volume of all samples decreases during incineration, for sample K the loss in volume is more substantial than in any other sample. In samples B , G , L and M, the change of colour due to the addition of additives can be noted. All samples containing beech wood develop similar colour patterns. Samples D, E, I, J, N, O, S and T all show a 2 colour pallet, with the harder outer mantle which had direct contact with the matrix being orange, while the rest of the pellet skeleton still retains the colour tone of the original sample.

4.3. Ash microscope analysis

4.3.1. Gathered data

Fig.40 shows the remains of samples B and S after the experiment is finished. Here, a round and relatively flat drop of molten ash forms. This structure can be considered the typical behaviour, similar results are also found in samples A, D, E, M, N, O, P and T. Those samples display varying degrees of homogeneity, with sample B having a relatively homogeneous colour palette, and sample S showing darker dots which indicate different thermochemical reactions taking place within the sample. When utilizing pellets consisting of fuel plus additive in reactors, such inhomogeneities may cause issues, since they could potentially lead to localised deviations from expected ash softening behaviours.

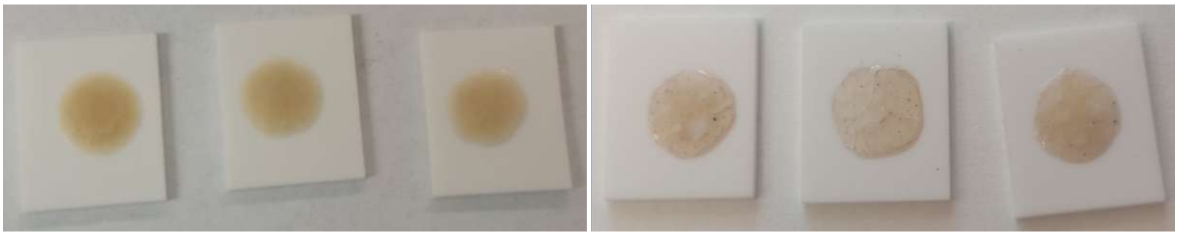


Fig. 40.: *Samples B (left) and S (right) after the experiment*

Depending on the feasibility of the different samples 2 or 3 experiments were conducted. According to the DIN EN ISO 21404 standardisation, only 2 representative samples are required, thus only if the gathered data is unclear, additional samples are necessary. The chosen naming pattern for samples is as follows, the letter indicates the used fuel and additive mix, while the number indicates the number of the repetition.

The ash softening and melting behaviour of samples B 3 and S 3 are shown in fig.41 fig.42. Again, those can be considered typical, since all relevant points of interest occur, which means shrinking, softening, spherical (in case of sample S 3), hemispherical and flow. Samples with such behaviour do generally not cause problems during the conduction of the experiment.

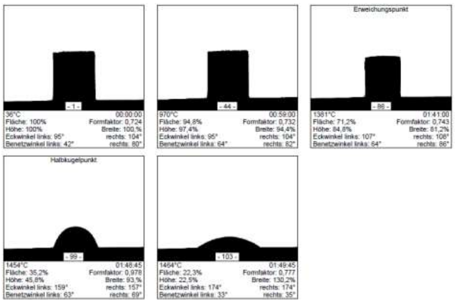


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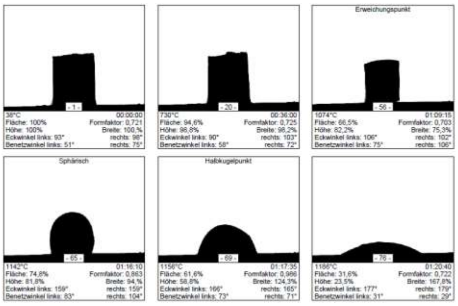
Fig. 41.: *Results of sample B 3*



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Benetzwinkel links: 31° rechts: 29°

Fig. 42.: *Results of sample S 3*

Sample F shows unusual behaviour during the experiment, the direct results can be seen in fig. 44. The spread of material indicates a volatile expansion of material during the heating. After the experiment, the sample plate was sticking to the sample carrier, which in turn required careful removal in order to not damage the carrier. This expansion can also be seen in fig. 43 and fig.44, both in form of an uneven spead on the plate and in form of dark fog/particles on the right side. Since this behaviour already occurs below 1200 °C in pure straw, and also occurs in straw/wood blends, those combinations seem to not be a viable foundation for pellets. Based on this further testing regarding the viability of straw be necessary. Similar behaviours were also seen in samples K and Q, with K also being below he required threshold, while Q being above it.

Fig. 43.: *Sample F after experiment*

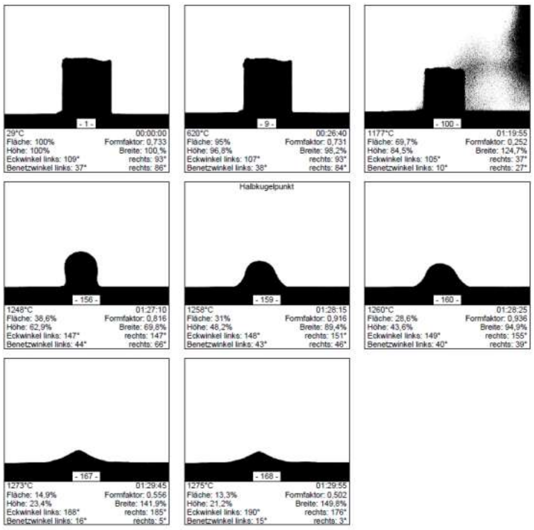

Fig. 44.: *Results of sample F 3*

Miscanthus ash shows substantial inflation during the ash microscope analysis, as can be seen in the third stage in fig. 45. According to DIN EN ISO 21404[7], inflation also counts as ash softening, this has to be taken into consideration when using miscanthus as fuel. This level of inflation did however only occur when using pure miscanthus, blends with wood did however also form spherical shapes. Based on the data alone, it cannot be clarified if this spherical shape is caused through inflation, which could indicate a lower ash softening temperature than suggested by the data. For clarification, further testing is required.

Fig. 46 shows the typical behaviour of all samples where no ash softening takes place, those are: G, H and R. All taken ash microscope pictures, as well as all gathered numerical data can be found in the appendix.

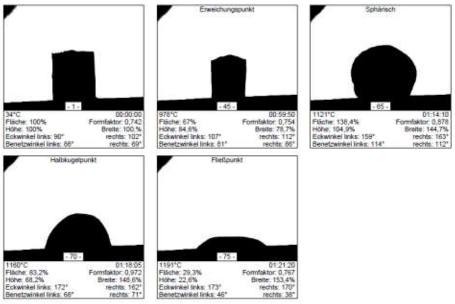

Fig. 45.: *Results of sample P 1*

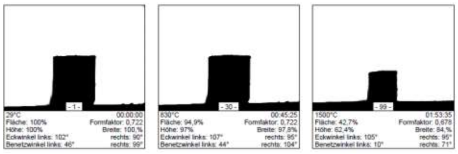

Fig. 46.: *Results of sample G 2*

4.3.2. Interpretation and analysis

The summarised results are shown in fig. 47 to fig. 51. Furthermore all collected data, both values and visual, can be found in the appendix A. Green bars hereby represent hay-samples, yellow bars straw-samples, blue bars corn cob samples and black bars miscanthus-samples, while the dark blue bar on top represents the maximum temperature of the sample chamber. Additionally, two lines are drawn in each diagram, one at 1100 °C and one at 1200 °C, those lines represent the minimum required values for usability. According to Lessig [6], am minimum SST of 1100 °C for ash softening is required to make pellets from non-woody biomass viable, for a guaranteed viability, an ash softening temperature of 1200 °C is the target. As stated in chapter 2.2.4, it is not clear whether the SST or the DT is the defining point for the usability of fuels in regards to the clogging of the gasifiers, therefore both points are looked at in depth. The calculated indices only estimate the SST, where no shape change takes place. The first point with visible shape change is the DT, based on that the target temperatures are to be looked at for all different points of ash softening behaviour, with special focus on SST and DT. Some bars are drawn hatched, this represents that only a single sample was used, filled bars on the other hand have been created from the mean of two or three samples, in accordance with the DIN EN ISO 21404[7] standardisation, which requires at least two samples. As a general rule the higher the temperature of every point of ash softening behaviour, the better. [6][7]

Primary Results

For this thesis, two points within the ash softening behaviour are of primary interest, the SST, on which the index calculations focus on, and the DT at which shape change fist appears.
Firstly, the results for the SST, which are shown in fig.47. According to DIN EN ISO 21404[7], the shrinking temperature is defined as the point, where the cross section area of the sample is 95 % of its original size, when looking at the results gathered from the ash microscope. Shrinking is the first step that occurs when heating up a sample. Samples C, P and Q are particularly interesting, since shrinking only takes place above the first threshold of 1100 °C, and in case of C and R even above 1200 °C.

When looking at the SST depicted in table 11, a slight correlation between additives can be seen. In general, the addition of wood leads to a slight increase of shrinking temperature, while the addition of inert materials leads to a somewhat larger increase.

The result do in some points align with the estimation done by [6], according tho which an SST above 1100 °C cannot be reached through the addition of kaolin. With 7.5 % lime, an SST even above 1200 °C is indeed possible, but only in case of hay and miscanthus. For dolomite, the miscanthus result even surpasses the expectations, while the straw result remains below the expected temperature.

Table 11.: *Experimentally determined SST*

	Mean SST of different fuel in °C			
Amount of additive	Hay	Straw	Corn cobs	Miscanthus
No additive	A: 720	F: 620	K: 610	P: 720
5% Kaolin	B: 970	n.a.	L: 900	n.a.
5% Dolomite	n.a.	G: 820	n.a.	Q: 1170
7.5% Lime	C: 1240	H: 700	M: 780	R: 1260
33% beech wood	D: 750	I: 600	N: 660	S: 730
66% beech wood	E: 800	J: 620	O: 670	T: 760

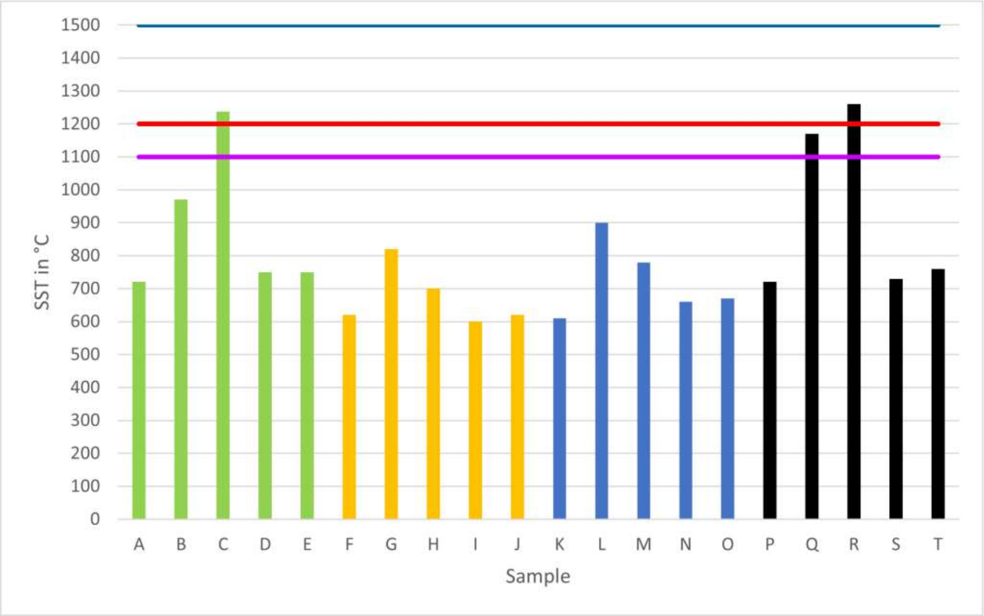

Fig. 47.: *SST of all samples*

The second main point of interest is the DT, which is visually depicted in fig. 48. The data gathered is shown in table12

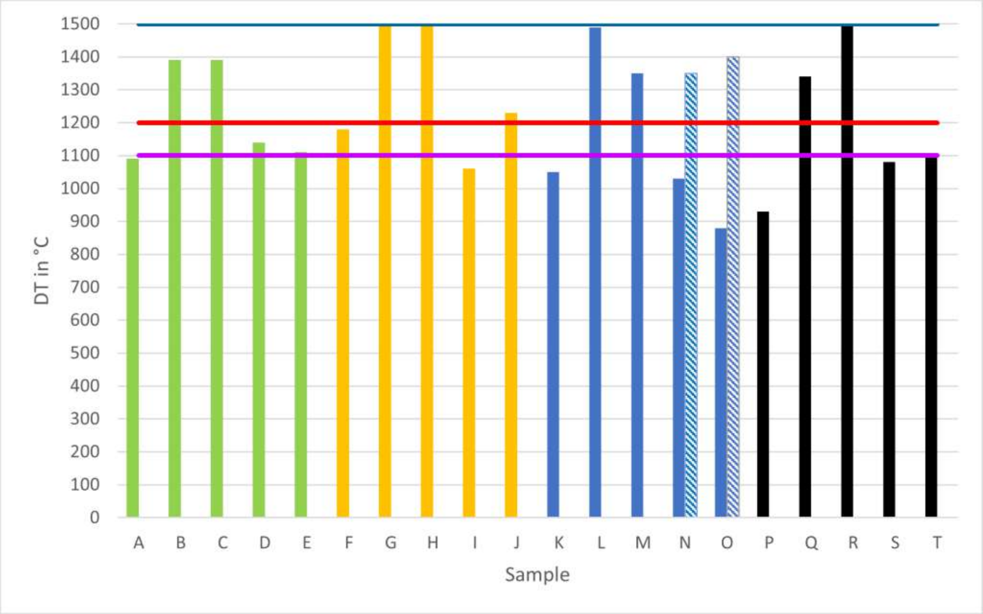

Fig. 48.: *DT of all samples*

Table 12.: *Experimentally determined DT*

	Mean DT of different fuel in °C			
Amount of additive	Hay	Straw	Corn cobs	Miscanthus
No additive	A: 1090	F: 1180	K: 1050	P: 930
5% Kaolin	B: 1390	n.a.	L: 1490	n.a.
5% Dolomite	n.a.	G: >1500	n.a.	Q: 1340
7.5% Lime	C: 1390	H: >1500	M: 1350	R: >1500
33% beech wood	D: 1140	I: 1060	N: 1030/1350	S: 1080
66% beech wood	E: 1110	J: 1230	O: 880/1400	T: 1100

To summarise, Samples

- B (95 % hay, 5 % kaolin)
- C (92.5 % hay, 7.5 % lime)
- G (95 % straw, 5 % dolomite)
- H (92.5 % straw, 7.5 % lime)
- J (33 % hay, 66 % beech wood)
- L (95 % corn cobs, 5 % kaolin)
- M (92.5 % corn cobs, 7.5 % kaolin)

- Q (95 % miscanthus, 5 % dolomite)
- R (92.5 % miscanthus, 7.5 % lime)

all produce results clearly above the required threshold. According to this data, those fuel and additive combinations should be viable for usage within biomass gasifiers, without the risk of ash meting and subsequent clumping within the gasifier. It has to be noted that sample J is only just above the threshold, therefore impurities, more heterogeneous additive distribution or other external factors may pushes the actual ash softening temperature below the threshold.
For samples

- N (66 % corn cobs, 33 % beech wood)
- O (33 % corn cobs, 66 % beech wood),

two possible temperatures were found, due to less clear data, as shown in fig. 85-86 and fig. 87-88 in the appendix A. Due to one value being above and one clearly below the threshold, no clear statement about the viability of those samples can be made, therefore further research is required.
Samples

- A (100 % hay)
- D (66 % hay, 33 % beech wood)
- E (33 % hay, 66 % beech wood)
- F (100 % straw)
- S (66 % miscanthus, 33 % beech wood)
- T (33 % miscanthus, 66 % beech wood)

are all very close to the lower threshold of 1100 °C. While insufficient by itself, further modification could also make those combinations viable.
Finally, Samples

- I (66 % straw, 33 % beech wood)
- K (100 % corn cobs)
- P (100 % miscanthus)

are clearly below the acceptable threshold.
A summary of all primary results is presented in table 13. Here it can be seen, that the calculated SST indices are not a viable estimate for the actual SST, they are however very much viable to asses the DT. According to this data, an index of 2 or lower almost always leads to a DT higher than 1200 °C, with only one exception in form of sample E. Even in this case though a DT above 1100 °C is reached. In some cases, namely J and L even an index

between 2 and 3 can lead to acceptable DT.

When comparing the expectations with the results, ash softening temperatures above 1200 °C can be achieved through the use of either 5 % kaolin, 5 % dolomite or 7.5 % lime in all tested samples, which would exceed the expectations of the index calculations if they are considered for the DT, according tho which this should have only been possible when using 7.5 % of either dolomite or lime. When looking at the also tested woody/non woody biomass blends, acceptable ash softening temperatures could only be achieved when using 33 % straw, combined with 66 % beech wood. For the corn cob/beech wood samples, ambiguous results were achieved, thus no clear statement regarding their viability can be made. Generally speaking though, the addition of wood did lead to an increase of ash softening temperature, using woody/non woody biomass blend with some inert additives could also be viable, for this further research is necessary.

Table 13.: *Calculated indices and related expected and calculated temperatures*

	Index values / temperatures in °C			
Sample	SST index	e. SST	m. SST	m. DT
A: Hay	2.49	≈1100	720	1090
B: Hay + 5% kaolin	1.85	1100-1200 °C	970	1390
C: Hay + 7.5% lime	1	>1200	1240	1390
D: Hay + 33% beech wood	2.03	≈1100	750	1140
E: Hay + 66% beech wood	1.57	1100-1200	800	1110
F: Straw	5.8	<1000	620	1180
G: Straw + 5% dolomite	1.22	1100-1200 °C	820	>1500
H: Straw + 7.5% lime	1.40	1100-1200 °C	700	>1500
I: Straw + 33% beech wood	4.24	<1000	600	1060
J: Straw + 66% beech wood	2.68	≈1100	620	1230
K: Corn cobs	5.76	<1000	610	1050
L: Corn cobs + 5% kaolin	2.95	≈1100	900	1490
M: Corn cobs + 7.5% lime	1.51	1100-1200	780	1350
N: Corn cobs + 33% beech wood	4.21	<1000	660	1030/1350
O: Corn cobs + 66% beech wood	2.66	≈1100	670	880/1400
P: Miscanthus	5.98	<1000	720	930
Q: Miscanthus + 5% dolomite	1.89	1100-1200	1170	1340
R: Miscanthus + 7.5% lime	1.47	1100-1200	1260	>1500
S: Miscanthus + 33% beech wood	4.36	<1000	730	1080
T: Miscanthus + 66% beech wood	2.74	≈1100	760	1100

Secondary Results

The ST is special in that spherical shapes do not occur with all samples, but only some. When looking at those samples, as displayed in fig. 49 and table 14 it can be noted that corn cobs have a lower tendency do create spherical shapes. For C, M and N only one out of three samples yielded a spherical shape, while for B, G, H, J, K, L, N, Q and R no spherical shape was found below the temperature maximum of 1500 °C. Spherical shapes could potentially appear at higher temperatures. Overall, when either kaolin or dolomite was added to a sample, no spherical shapes formed.

Table 14.: *Experimentally determined ST*

	Mean ST of different fuel in °C			
Amount of additive	Hay	Straw	Corn cobs	Miscanthus
No additive	A: 1120	F: 1250	K: n.a.	P: 1110
5% Kaolin	B: n.a.	n.a.	L: n.a.	n.a.
5% Dolomite	n.a.	G: n.a.	n.a.	Q: n.a.
7.5% Lime	C: 1325	H: n.a.	M: 1328	R: n.a.
33% beech wood	D: 1240	I: 1320	N: n.a.	S: 1140
66% beech wood	E: 1140	J: n.a.	O: 1424	T: 1130

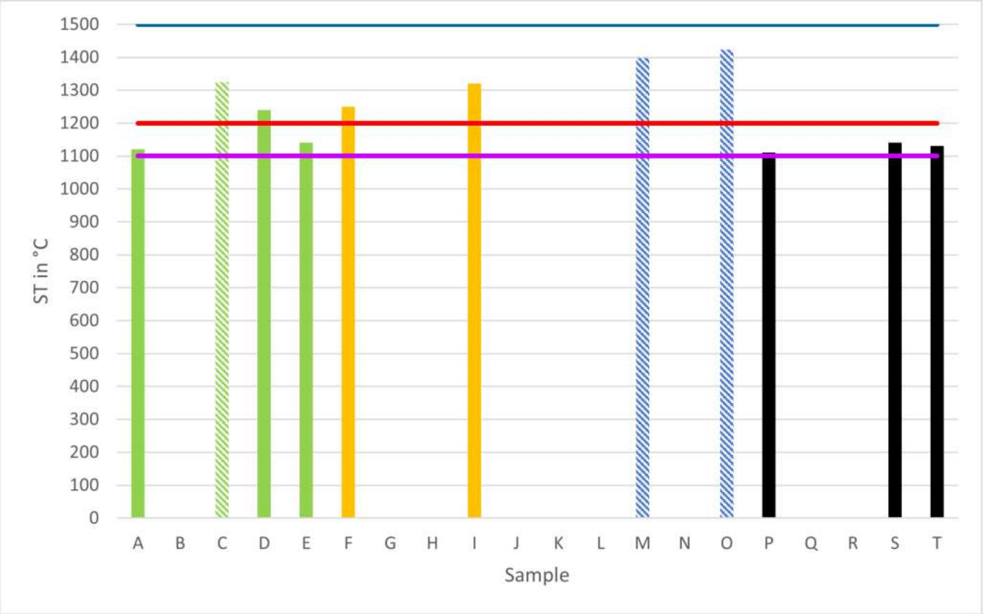

Fig. 49.: *ST of all samples*

Fig. 50 and table 15 show the HT. For Samples C, G, H, L and R this shape was not achieved, within the temperature framework, it therefore has to be above 1500 °C. Hay and miscanthus show relatively low values, both in pure form and when blended with wood, while the temperatures of straw and corn cobs are relatively high. For any corn cob mixture, the gathered values are relatively close to each other.

Table 15.: *Experimentally determined HT*

	Mean HT of different fuel in °C			
Amount of additive	Hay	Straw	Corn cobs	Miscanthus
No additive	A: 1140	F: 1260	K: 1330	P: 1150
5% Kaolin	B: 1460	n.a.	L: >1500	n.a.
5% Dolomite	n.a.	G: n.a.	n.a.	Q: 1370
7.5% Lime	C: 1342	H: n.a.	M: 1450	R: n.a.
33% beech wood	D: 1260	I: 1400	N: 1480	S: 1160
66% beech wood	E: 1160	J: 1330	O: 1480	T: 1140

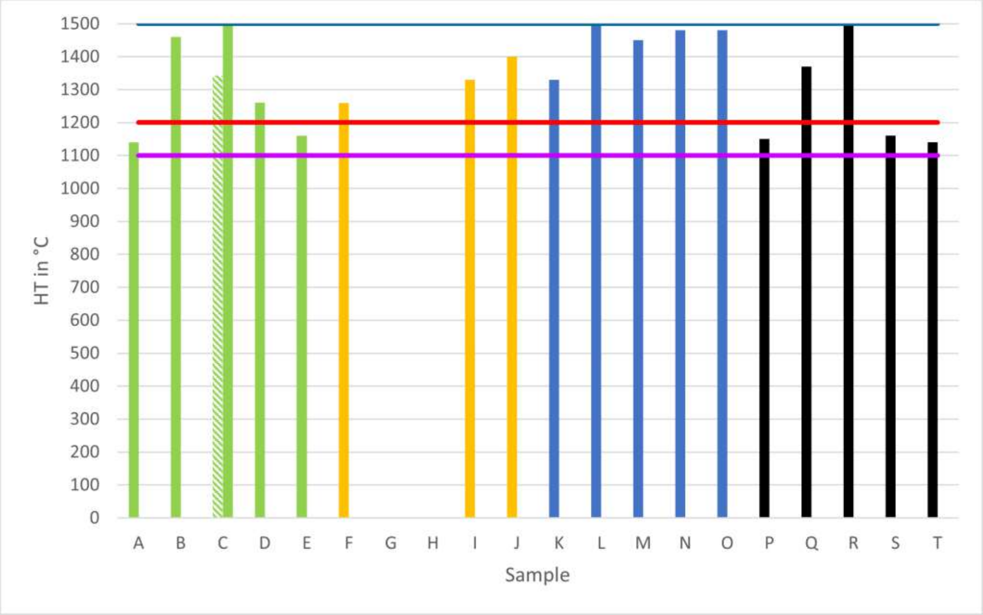

Fig. 50.: *HT of all samples*

The FT of all samples are shown in fig. 51 and table 16. Samples G, H, L and R do not flow even at the maximum temperature of 1500 °C, while samples B, N and O flow around the 1500 °C mark. The flow temperature is lower than 1200 °C for samples A, E, P, S and T. In summary, all samples which use either kaolin, dolomite ore lime have a flow temperature above 1200 °C, while all samples with a flow temperature below 1200 °C are either pure hay/miscanthus or hay/miscanthus mixed with beech wood. All straw and corn cob samples display a flow temperature above 1200 °C.

Table 16.: *Experimentally determined FT*

	Mean FT of different fuel in °C			
Amount of additive	Hay	Straw	Corn cobs	Miscanthus
No additive	A: 1160	F: 1270	K: 1370	P: 1170
5% Kaolin	B: 1470	n.a.	L: >1500	n.a.
5% Dolomite	n.a.	G: >1500	n.a.	Q: 1390
7.5% Lime	C: 1362/>1500	H: >1500	M: 1460	R: >1500
33% beech wood	D: 1280	I: 1340	N: 1490	S:1190
66% beech wood	E: 1190	J: 1420	O: 1490	T: 1160

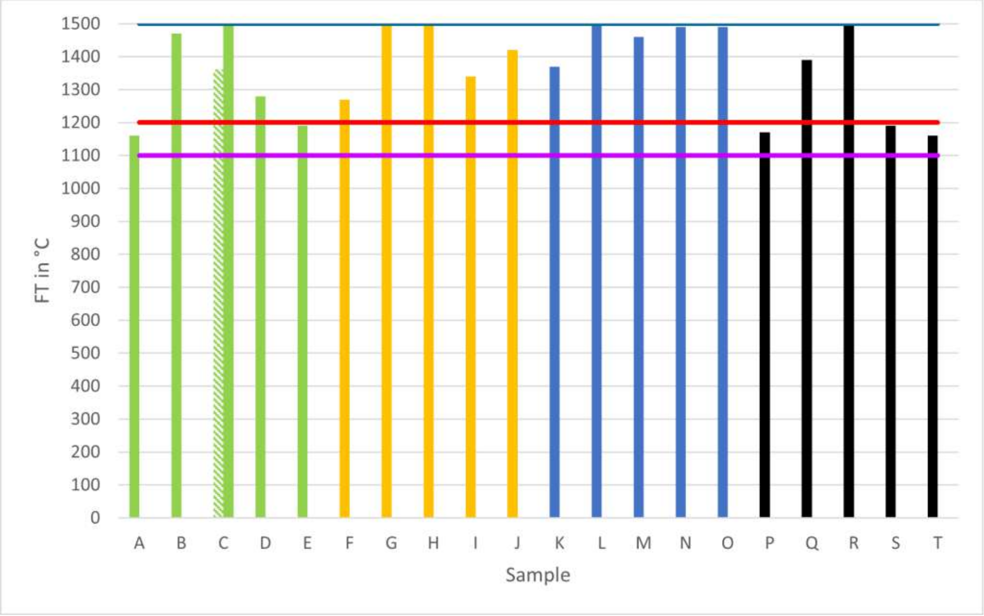

Fig. 51.: *FT of all samples*

Comparison

Diem[34] also tested woody and non-woody biomass with and without additives through the ash microscope. In particular, the influence of the incineration temperature on the pellets and the ash softening behaviour was tested. Overall, the main results were:

- The ash softening of woody biomass starts above 1400 °C.
- Non wood-biomass has generally low ash softening temperatures, often below 1000 °C
- A change of incineration temperature dos not lead to any significant change in ash softening behaviour.
- The addition of additives leads to an increase of ash content, in case of kaolin up to 58 %, for lime up to 25 %.

When comparing these results to this thesis, the following conclusions can be drawn:

- No pure woody-biomass samples were tested, thus no direct comparisons can be made. In this thesis however, blends from woody and non-woody biomass were tested, the ash softening behaviour of those was generally more similar to the non-woody biomass and thus comparatively low.
- The ash softening temperatures of non-woody biomass without additives ware also around 1000 °C and below the threshold of 1200 °C, make the use of additives or other pre-treatment techniques mandatory.
- All incineration was done using the same temperature program, so no statement can be given regarding the effects of incineration temperature.
- An increase of ash content through the use of additives wasn't directly measured, but visual analysis, as seen in fig.36, fig.37, fig.38 and fig.39, also shows an increase.

5. Conclusion and Outlook

5.1. Conclusion

To summarise, pellets from pure non-woody biomass, as well as biomass/additive blends were produced. The production of serviceable pellets was made possible through shredding the material into fitting size, followed by pelleting with the addition of water, and the utilisation of multiple passages. All tested combinations led to serviceable pellets, drying after pelletisation is however necessary to prevent mould. Pellets made from harder materials, such as corn cobs or non-woody/woody biomass blends have a tendency to clog the pellet press, further fine-tuning is necessary to ensure proper throughput. The pellets were incinerated and the resulting ash subsequently analysed via an ash microscope to determine the ash melting behaviours, in particular the ash softening temperature, as described by the EN ISO DIN 21404 standardisation. The addition of either 5 % kaolin, 5 % dolomite or 7.5 % lime all lead to serviceable ash melting behaviours above 1200 °C, which even exceeded the expected temperatures from index calculations. The combination of 33 % straw and 66 % beech wood also produced values above the required threshold, although only narrowly. For the blends of corn cobs and beech wood, the results were unclear, further testing is required to determine their viability. The combined main results are summarised in table13. It must also be noted that the testing in the ash microscope may not fully represent the actual behaviours as the changing conditions within gasifiers may lead to different outcomes, thus testing in pilot plants is required. For further improvements, techniques such as torrefication, biochar or even just fine tuning of blend composition may be used. With woody biomass being a continued subject of criticism regarding its climate balance, non woody-biomass may serves as a powerful alternative, even though additional development is still required.

When looking at the research questions, they can be answered as follows:

- Can the theoretically investigated enhancements of ash melting temperatures for non-woody biomass fuels mixed with additives be confirmed experimentally?
  - The enhancements of the SST cannot be confirmed experimentally, the enhancements of the DT however can be confirmed. According to the gathered data, an index of 2 or lower leads to the target DT of 1200 °C or higher.
- Does the estimation of the ash melting behaviour through the calculation of fuel indices provide a sufficient accuracy?

  - In accordance with the first research question, for the DT the connections between the calculated index and the measured DT can be considered sufficient. Three outliers occur, in two cases the DT is higher than expected, and only in one case, sample E, the measured DT is lower.

- Could the use of additives in non-woody biomass fuels for CHP application with fixed bed gasification be a technical, ecological and economical alternative to woody biomass?
  - From a technical point of view, pellets made from non-woody biomass with additives deliver serviceable results, both in regards to physical properties and fuel properties. The annual or perennial growth of those non-woody plants is, ecologically speaking preferable to the long growth times of trees. Finally, from an economic point of view, such pellets could be a viable alternative, if local availability and transport costs a suitable.

5.2. Outlook

While the results of this thesis indicate a general usability of pellets based on non-woody biomass, in particular those with inert materials added, the ash microscope analysis alone is insufficient to fully asses their viability. The ash microscope has a generally not moving atmosphere within its sample chamber, which is not true for gasifiers. In fixed bed gasifiers, material moves downward through the reactor, while in fluidized bed the whole material is moving. The separation of fuel material and additive through this movement, especially when under the effect of thermochemical conversion poses is significant risk. This makes further experimentation, in particular in small scale gasifiers with actual process conditions, necessary. The effectiveness of the pellets and separation risks could also be influenced by the grain size distribution of the additive and fuel material. A future research project may analyse those factors in order to optimize potential of such pellets blends.
While the use of inert additives such as lime has yielded positive results, they are comparatively expensive. Combining both inert additives, woody and non woody biomass may be examined for more cost effective yet still serviceable pellets.

While pure non-woody biomass pellets have generally shown unfavorable results, pre-treatment methods and specific process parameters may positively influence them. Some potential methods are presented here:

[43] analysed pure and blended pellets with pine sawdust as base material in order to find the ideal pelletisation process temperature and moisture content. Furthermore, the energy consumption when using different kinds and amounts of blend material was determined. This was done through the use of response surface methodology (RSM). The following results were achieved: The ideal process temperature was 80 °C, with a moisture content of 16.6 %. All other relevant parameters were within the limitation set by the DIN EN ISO 17 225-2

standardisation. The energy consumption of pure pine sawdust pellets was 0.18 kWh/kg, while with blends the consumption ranges between 0.09 and 0.33 kWh/kg. Blends using cocoa shells, coffee dregs, coffee heaps, grape pomance and miscanthus had a lower energy consumption than pure pine sawdust pellets.

Widjaya et at. [68] summarised several other techniques which may also be used to improve the viability of non-woody biomass. On one hand, torrefication, which can be described as drying of the feed material at 200 to 300 °C in inert atmosphere, could be used as an alternative to pelletisation. Another alternative pre-treatment is the creation of biochar through hydrothermal carbonisation, which could subsequently be used as a foundation for pellets. It has to be noted though, that fine-tuned pellet compositions may only be relevant in a last step before market introduction of such pellets, as further and more basic research is necessary to create a serviceable foundation.

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A. General Appendix

A.1. Data

Table 17.: *Overview of all collected data*

	Shrinking	Softening	Spherical	Hemispherical	Flow
Sample	temperature in °C				
A1	718	1080	1123	1140	1162
A2	724	1099	1126	1142	1162
A3	721	1080	1125	1142	1161
A mean	720	1090	1120	1140	1160
B1	964	1394	n.a	1462	1471
B2	980	1399	n.a	1456	1465
B3	970	1381	n.a	1454	1464
B mean	970	1390	n.a	1460	1470
C1	1311	1366	n.a.	n.a.	n.a.
C2	1302	>1500	n.a.	n.a.	n.a.
C3	1103	1311	1325	1342	1362
C mean	1238	1390	1325	1340	1362
D1	766	1132	1243	1269	1289
D2	753	1140	n.a.	1252	1278
D3	739	1135	1230	1252	1272
D mean	750	1140	1240	1260	1280
E1	766	1122	1160	1181	1211
E2	753	1120	1164	1188	1217
E3	739	1098	1102	1116	1146
E mean	750	1110	1140	1160	1190
F1	623	1178	1250	1259	1270
F2	614	1187	1260	1266	1278
F3	622	1177	1248	1259	1274
F mean	620	1180	1250	1260	1270
G1	815	n.a.	n.a.	n.a.	n.a.
G2	828	n.a.	n.a.	n.a.	n.a.
G mean	820	n.a., >1500	n.a.	n.a.	n.a.
H1	705	n.a	n.a.	n.a.	n.a.

H2	697	n.a	n.a.	n.a.	n.a.
H mean	700	n.a. >1500	n.a.	n.a.	n.a.
I1	597	1088	1318	1326	1344
I2	597	1041	1317	1330	1347
I mean	600	1060	1320	1330	1340
J1	633	1230	n.a.	1402	1424
J2	608	1238	n.a.	1396	1424
J mean	620	1230	n.a.	1400	1420
K1	611	1059	n.a.	1320	1356
K2	614	1057	n.a.	n.a.	n.a.
K3	593	1048	n.a.	1330	1386
K mean	610	1050	n.a.	1330	1370
L1	895	1480	n.a.	n.a.	n.a.
L2	899	1493	n.a.	n.a.	n.a.
L mean	900	1490	n.a.	n.a.	n.a.
M1	776	1377	n.a.	1455	1463
M2	787	1356	1398	1451	1460
M mean	780	1350	1400	1450	1460
N1	656	1000/1330	n.a.	1480	1489
N2	661	1050/1360	n.a.	1481	1486
N mean	660	1030/1350	n.a.	1480	1490
O1	58/675	850/1410	1424	1487	1497
O2	656	905/1390	n.a.	1468	1490
O mean	670	880/1400	1420	1480	1490
P1	723	940	1121	1160	1191
P2	722	940	1117	1153	1188
P3	719	900	1088	1133	1144
P mean	720	930	1110	1150	1170
Q1	1177	1353	n.a	1373	1389
Q2	1160	1338	n.a	1366	1366
Q3	1180	1326	n.a	1376	1392
Q mean	1170	1340	n.a	1370	1390
R1	1143	n.a.	n.a	n.a.	n.a.
R2	1253	n.a.	n.a	n.a.	n.a.
R3	1394	n.a.	n.a	n.a.	n.a.
R mean	1260	n.a., >1500	n.a	n.a.	n.a.
S1	728	1078	1137	1156	1182
S2	723	1087	1141	1160	1196
S3	728	1074	1142	1156	1186

S mean	730	1080	1140	1160	1190
T1	754	1089	1128	1139	1158
T2	748	1110	1131	1144	1162
T3	762	1113	1133	1149	1164
T mean	760	1100	1130	1140	1160

A.2. Datasheets

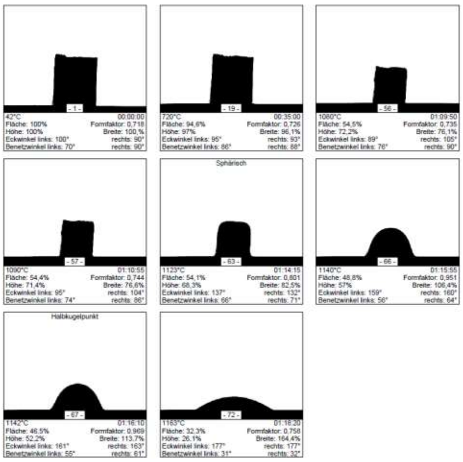


Fig. 52.: *Results of sample A 1*

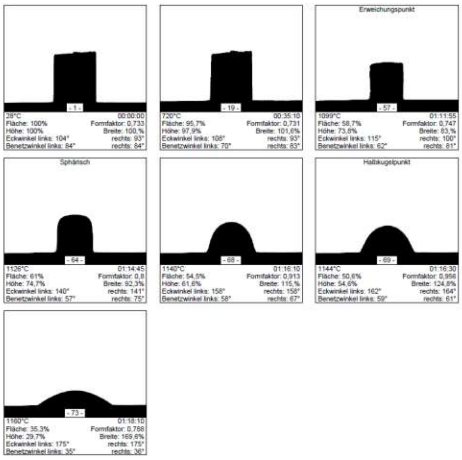


Fig. 53.: *Results of sample A 2*

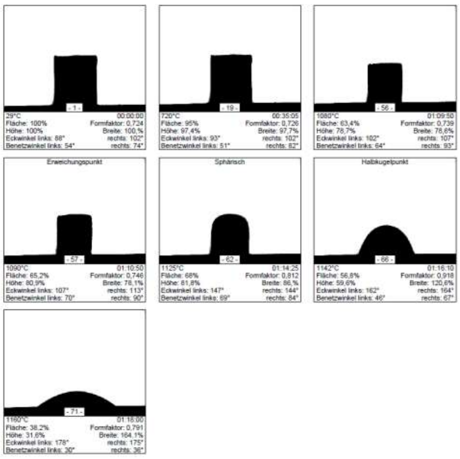


Fig. 54.: *Results of sample A 3*

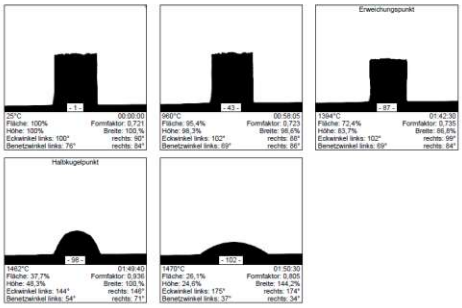


Fig. 55.: *Results of sample B 1*

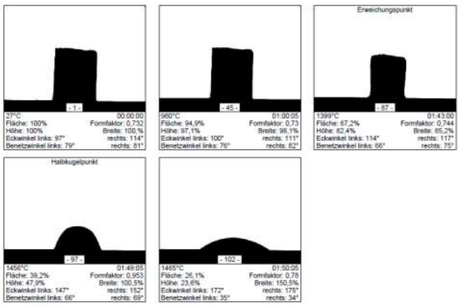


Fig. 56.: *Results of sample B 2*

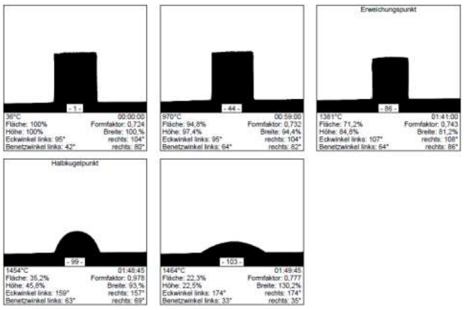


Fig. 57.: *Results of sample B 3*

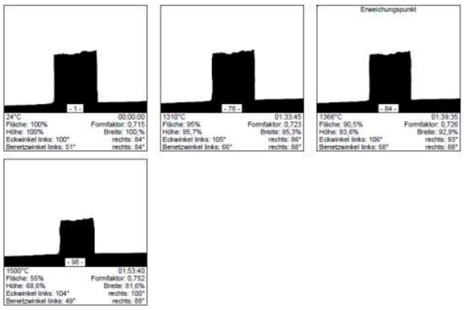


Fig. 58.: *Results of sample C 1*

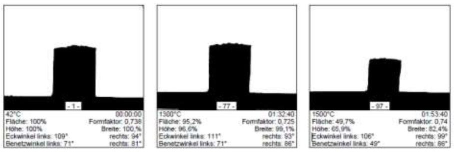


Fig. 59.: *Results of sample C 2*

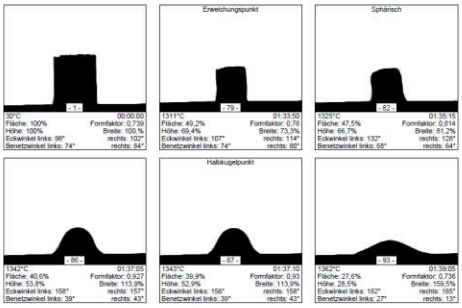
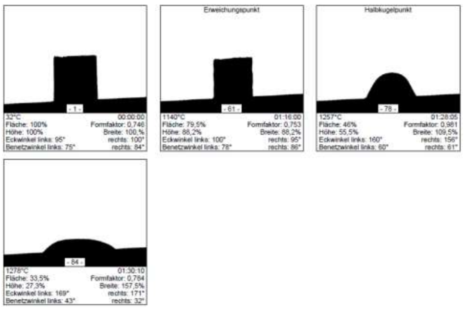


Fig. 60.: *Results of sample C 3*



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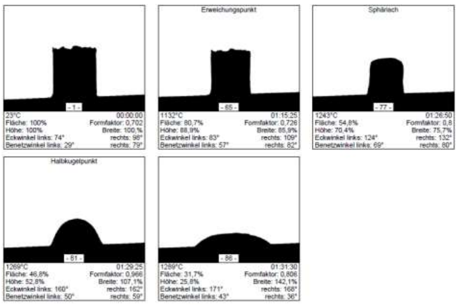
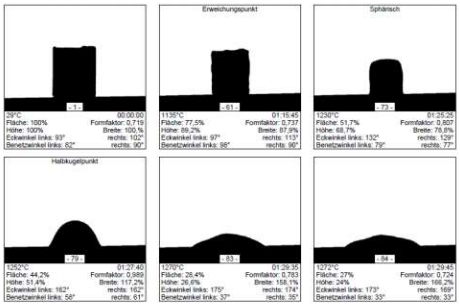


Fig. 62.: *Results of sample D 2*



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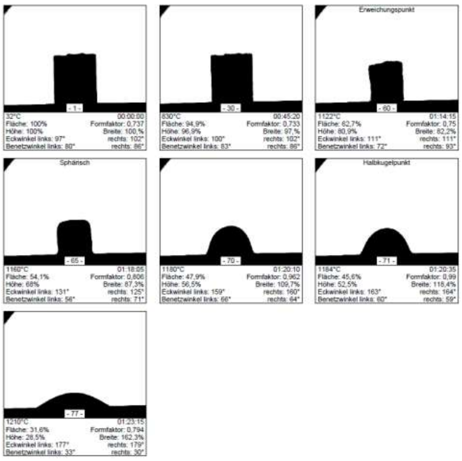
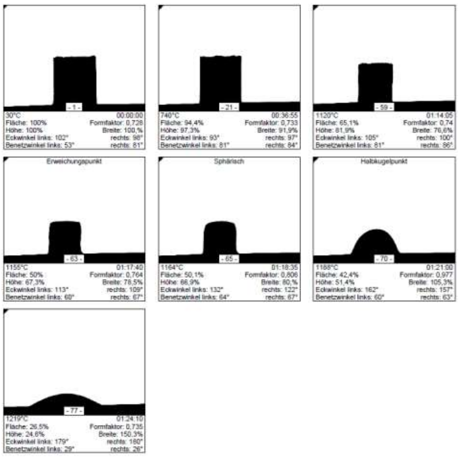


Fig. 64.: *Results of sample E 1*



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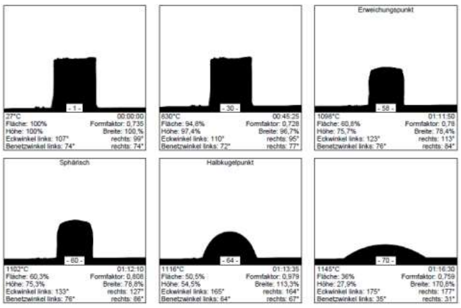


Fig. 66.: *Results of sample E 3*

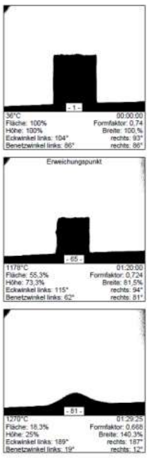


Fig. 67.: *Results of sample F 1*

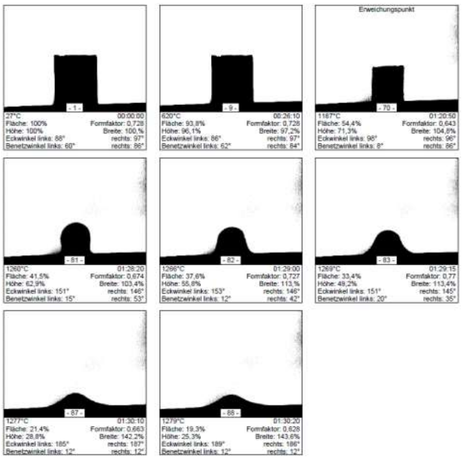
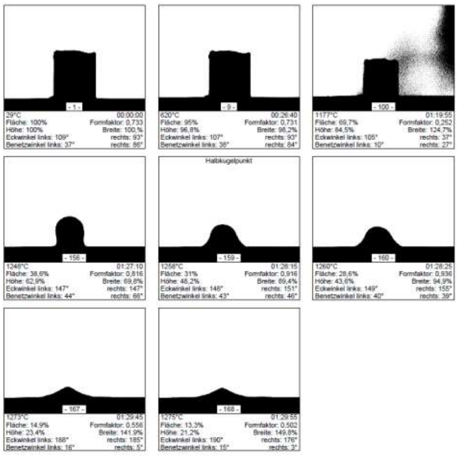


Fig. 68.: *Results of sample F 2*



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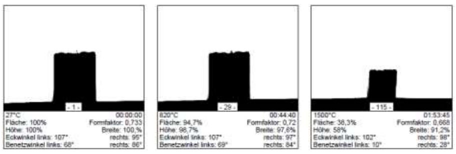


Fig. 70.: *Results of sample G 1*

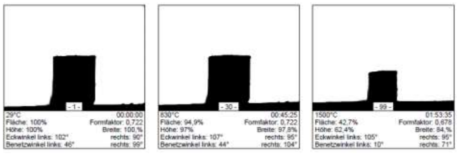
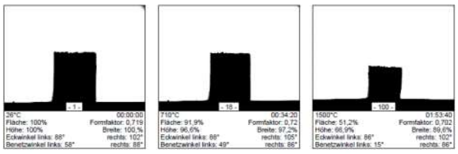


Fig. 71.: *Results of sample G 2*



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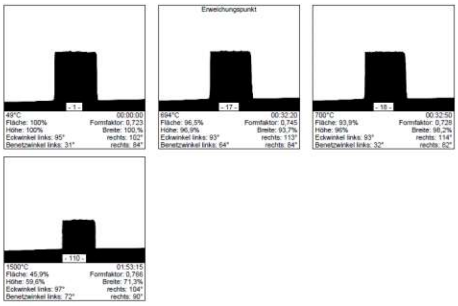


Fig. 73.: *Results of sample H 2*

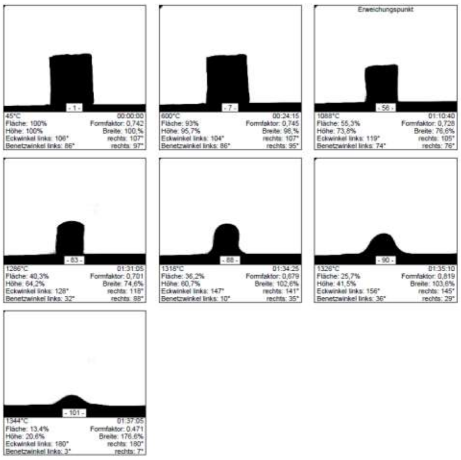


Fig. 74.: *Results of sample I 1*

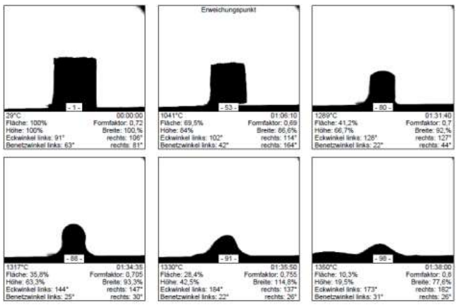


Fig. 75.: *Results of sample I 2*

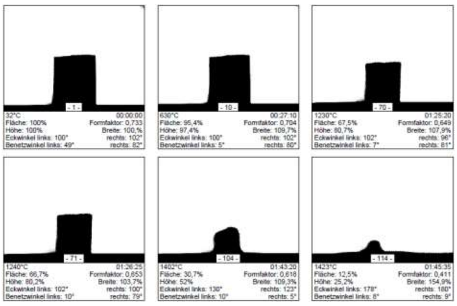


Fig. 76.: *Results of sample J 1*

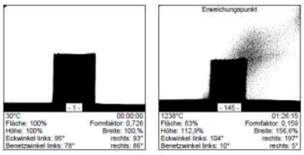


Fig. 77.: *Results of sample J 2*

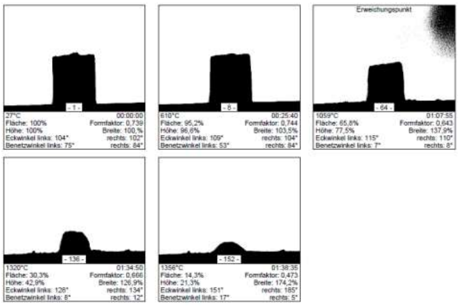
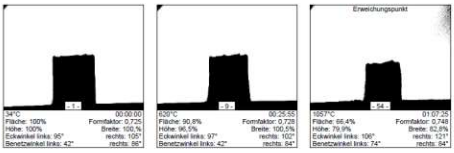


Fig. 78.: *Results of sample K 1*



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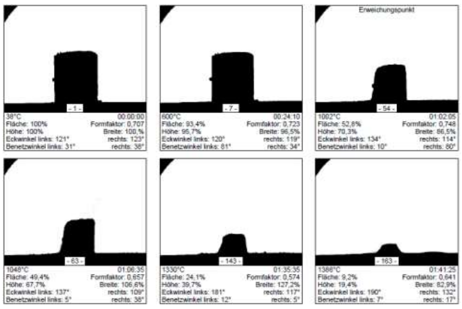

Fig. 80.: *Results of sample K 3*

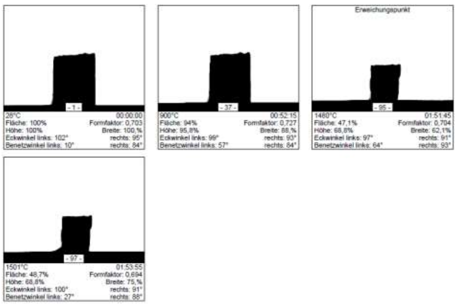

Fig. 81.: *Results of sample L 1*

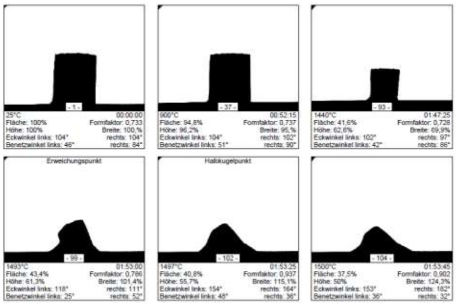

Fig. 82.: *Results of sample L 2*

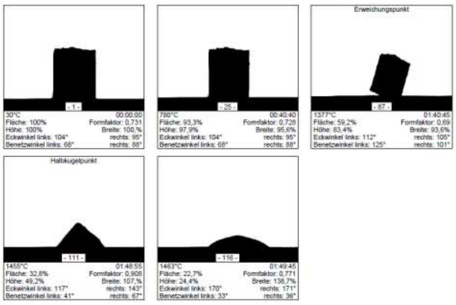

Fig. 83.: *Results of sample M 1*

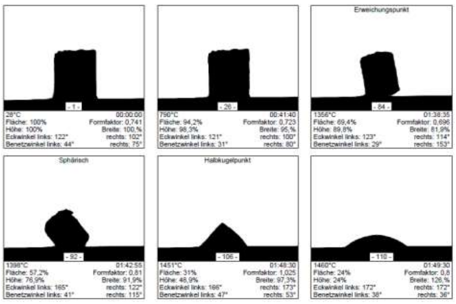

Fig. 84.: *Results of sample M 2*

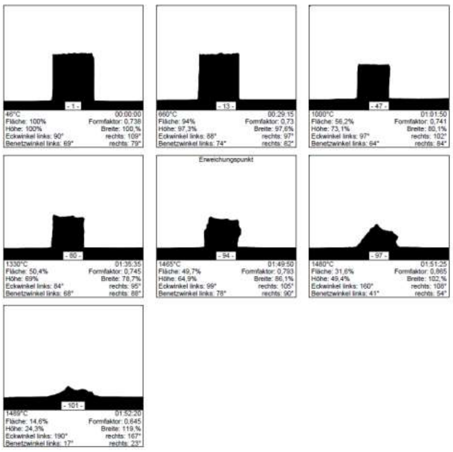

Fig. 85.: *Results of sample N 1*

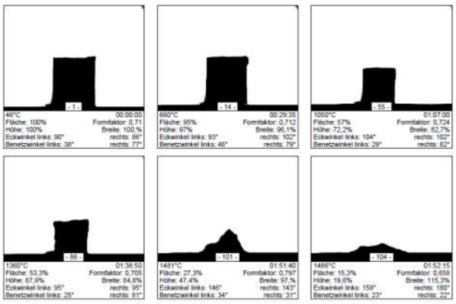

Fig. 86.: *Results of sample N 2*

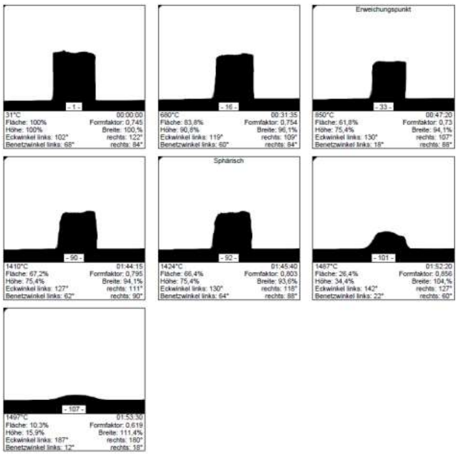

Fig. 87.: *Results of sample O 1*

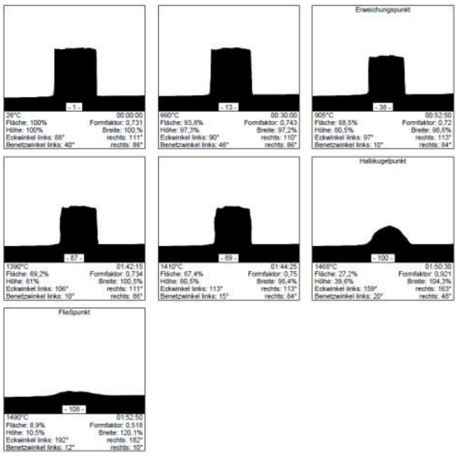

Fig. 88.: *Results of sample O 2*

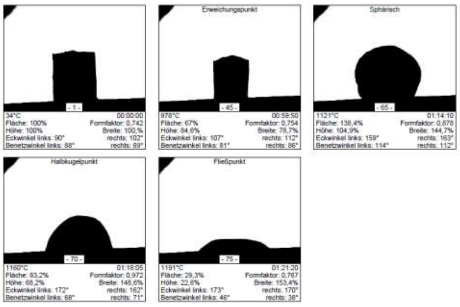

Fig. 89.: *Results of sample P 1*

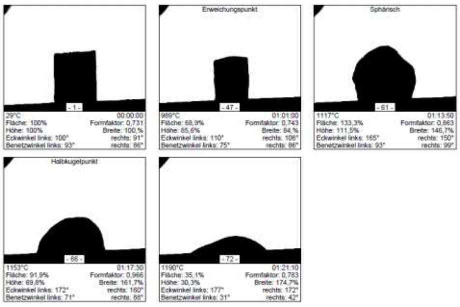

Fig. 90.: *Results of sample P 2*

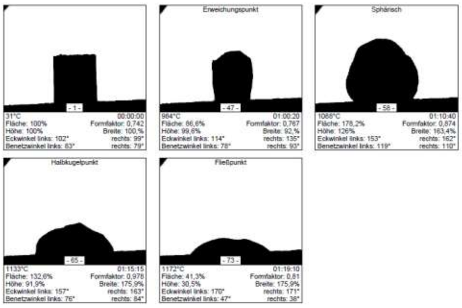

Fig. 91.: *Results of sample P 3*

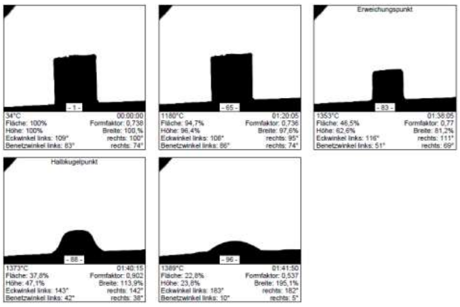

Fig. 92.: *Results of sample Q 1*

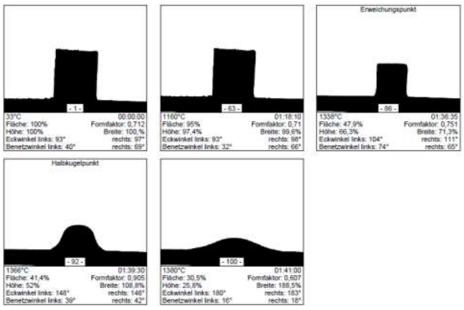

Fig. 93.: *Results of sample Q 2*

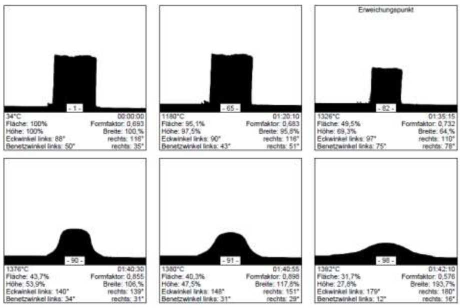

Fig. 94.: *Results of sample Q 3*

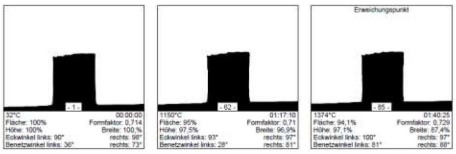

Fig. 95.: *Results of sample R 1*

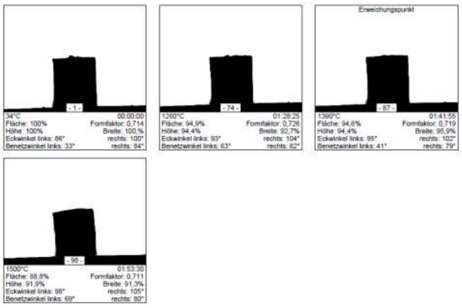

Fig. 96.: *Results of sample R 2*

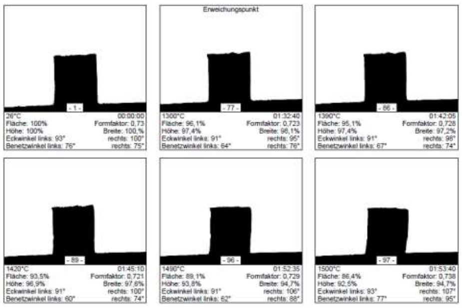

Fig. 97.: *Results of sample R 3*

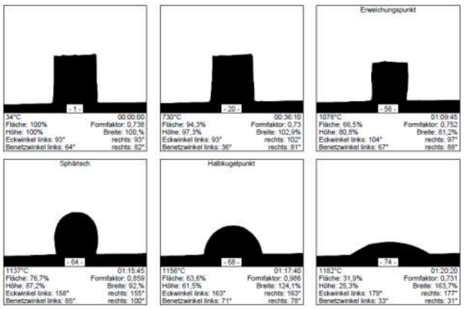

Fig. 98.: *Results of sample S 1*

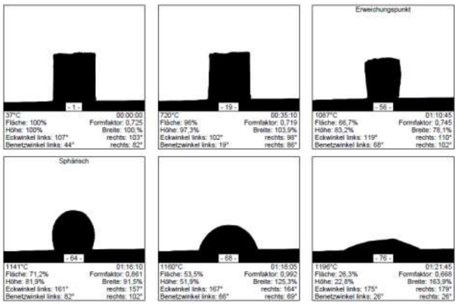

Fig. 99.: *Results of sample S 2*

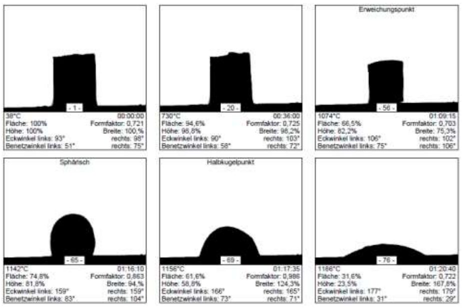

Fig. 100.: *Results of sample S 3*

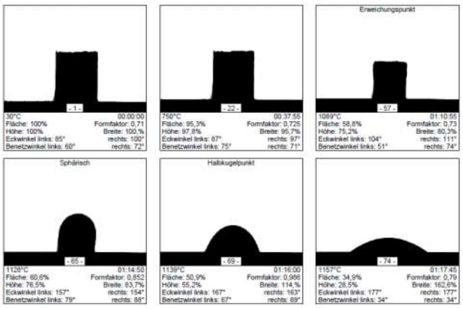

Fig. 101.: *Results of sample T 1*

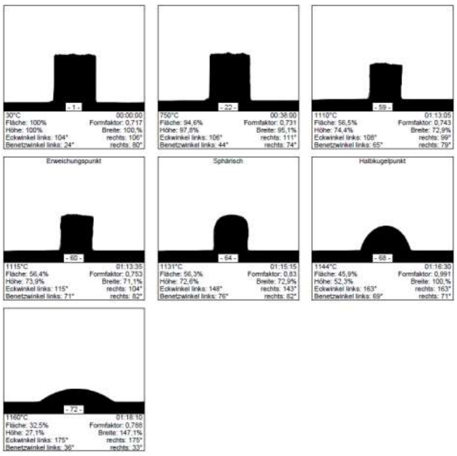

Fig. 102.: *Results of sample T 2*

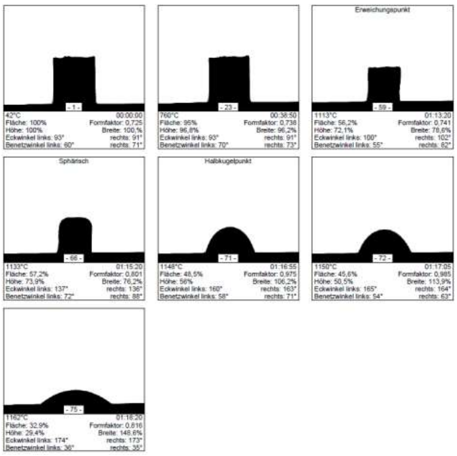

Fig. 103.: *Results of sample T 3*