Influence factors on the comminution process of wood for the production of precursors and basic chemicals for the chemical industry

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Abstract

The upstream process of comminution is a key element in the use of renewable raw materials, which impacts the consecutive disintegration of the materials. Energy efficiency of the comminuition process is therefore of utmost importance. The key factors to increase energy efficiency are, beside the mill type and the mill operation factors, the species of the renewable resource, in terms of water contend and the mechanical properties which are the dominant factors in biomass size reduction. In this study the influence of different factors on the effective specific comminution energy (ESCE) is elucidated. For theses purpose, three types of raw wood chips as well as recycled wood of two different qualities, were comminuted with a cutting mill and a swing hammer mill. The materials were comminuted at several levels of moister contend, under varied opening sizes of the internal screen of the mills. Particle size distributions of the comminuted materials were examined with sieve analyses and dynamic image analysis. Especially, the moister content directly influences the ESCE and the particle size distribution. Moreover the type of material, due to its different mechanical properties, is of significant influence on the particle size distribution and the ESCE. Copyright © 2019 VBRI Press.

Keywords: Wood, hammer mill, cutting mill, specific comminution energy, particle size.

Introduction

Renewable resources have been gaining increasing importance for energy generation in recent years. As a result of the development of innovative technologies, renewable raw materials are progressively being used for the production of precursors and basic chemicals for the chemical industry. Lignocellulosic biomass, as an alternative to limited crude oil, can be utilized to produce these chemicals. Materials such as dedicated crops (miscanthus, switchgrass), agricultural residues (corn stover, wheat straw) and forest products (hardwood and softwood) as well as recycling materials are suitable for the production of energy and basic chemicals. These biogenic materials generate low net greenhouse emissions and are sufficiently abundant. With the increasing demand for liquid biofuels such as bioethanol, which are produced from food crops like sugar cane or cereal crops, lignocellulosic materials provide an alternative feedstock. They consist of approximately 90 % cellulose, hemicellulose and lignin in dry mass [**1**]. The upstream process of comminution is a key element in the use of renewable raw materials, that impacts the consecutive disintegration of the materials, which means the separation of the three main components: cellulose, hemicellulose and lignin [**2-4**]. Comminution of lignocellulosic biomass is a crucial step in the process of utilizing these materials for the production of biochemicals, as it (I) decreases the cellulose crystallinity, (II) increases the accessible specific surface, (III) increases the number of pores, as well as their size and (IV) reduces the mass transfer limitation during the disintegration [**5, 6**]. Furthermore, comminution has an influence on the structure of lignin which might enhance the conversion rates of the following process [**7**]. It also plays a major role when these materials are used as solid biofuels for co-firing with coal or for combustion with pulverized burners [**8, 9**]. Furthermore, comminution is a required pre-treatment for the production of pellets [**10**]. Furthermore the yield of lignin extraction can be improved by a targeted comminution [**11**]. The conversion of lignocellulosic biomass into basic chemicals requires a fine comminution to improve the accessibility of reactive agents and enhance the conversion rates and yields. According to Bitra *et al.* biogenic raw materials have to be comminuted to around 1-6 mm for the ethanol production [**12**]. However, comminution is a highly energy consuming process, making it necessary to assess the benefits of a fine comminution in terms of reducing energy consumption by an adequate characterization of the products. Energy efficiency of the comminution that achieves the required particulate properties for subsequent processes is therefore of the utmost importance to make products from renewable resources more competitive with petrochemical products [**4, 13, 14**]. The key factors influencing the energy efficiency are, besides the mill type and the mill operation factors, the species of the renewable resource, in terms of water content and the mechanical properties which are the dominant factors in biomass size reduction. A better understanding of these interdependencies can help improve the adjustment of particle size distribution and particle shape. As well as the total specific surface area and the energy demand for the comminution process, which impacts the overall efficiency of the supply chain process, disintegration as well as the conversion. Furthermore, little research appears to have been done overall on the fine comminution of biomass as well as the characterization of the products. Miao *et al.*[**15**] showed that hammer mills, cutting mills and disc mills are the best-suited mills for biomass comminution. Hammer mills are the most popular commercially used devices applied to biomass comminution, so most research has been done on this type of mill. This work, therefore, focuses on the comminution of woody biomass in a cutting mill. It reports the effects of the influencing parameter of the biomass on the comminution process, the energy consumption and the physical properties of the products regarding their particle size. Furthermore, selected experiments have been carried out on a swing hammer mill to compare the influence of the type of stressing.

Experimental

Experimental setup and procedure

The comminution experiments were carried out on a cutting mill of the type Retsch SM 2000. Cutting mills are especially suitable for the comminution of fibrous and elastic materials due to the fact that cutting stress is the predominating force. The rotational speed of the mill was 750 min^{-1} which leads to a circumferential top speed of 5 m/s and the electric motor developed a rated power of 1500 W. The mill was equipped with 18 indexable tips and 4 fixed stator bars consisting of hard alloy. Four different internal screens where used for the purpose of the experiments. The screens were perforated with square meshes of the diameter 4, 6, 8 and 10 mm. Furthermore, comparative experiments, to study the influence of the kind of stress, were carried out selectively on a swing hammer mill. The main kind of stress in a hammer mill is stress through impact, whereas in a cutting mill it is cutting stress. A swing hammer mill of the type CONDUX LHM 20/60 with a rotational speed of 3600 1/min and a circumferential tip speed of 36 m/s was used. 28 rectangular hammers with a weight of 85 g comminute the material and an internal screen of 4 mm mesh width, determines the particle size distribution of the product. An outline diagram of the equipment used in this study is shown in **Fig. 1**. The mills were fed with a constant mass flow of $\dot{m}$ = 0.1 kg/min to avoid an overloading of the mills. The product was discharged in a box from which the products were collected to determine their particle size distributions.

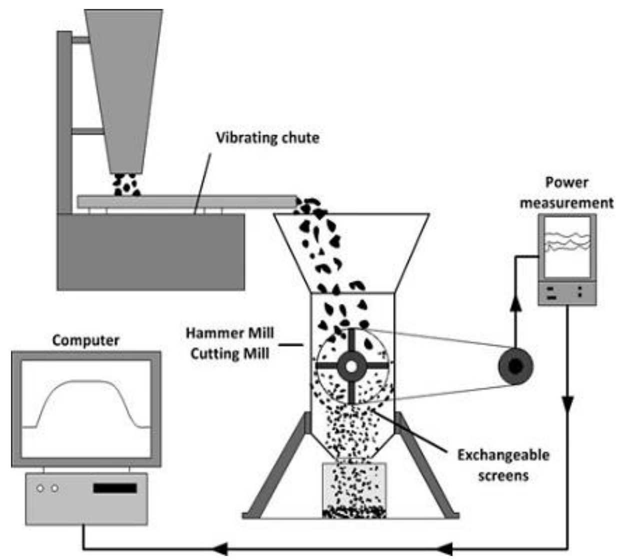

Fig. 1. Experimental comminution equipment.

In the present study, each experiment was repeated three times. The internal screen of the hammer mill was kept at 4 mm mesh widths and the ones for the cutting mill where varied between 4, 6, 8 and 10 mm. The internal screen of the mill determines at which particle size the particles get discharged from the mill. Furthermore, it is co-responsible for the residence time of the particles in the mill as well as how often a particle gets stress before it is discharged. At the start of each experiment, the no-load power consumptions of the mills were measured for 10 minutes and immediately afterward the comminution process took place. The power consumption of the mills was measured with the Fluke Power Quality and Energy Analyzer meter. With the no load power consumption and the total power consumption during the comminution process, the specific comminution energy can be calculated. The specific comminution energy E_{spec} can be calculated as shown in Equation (1) where m is the comminuted mass, P_t the total power consumption during the comminution and P_l the no-load power consumption [**16**].

$$E_{spec} = \frac{1}{m}\int_{t_0}^{t}(P_t - P_l)dt \qquad (1)$$

Characterization

The characterization of the particle size distributions was carried out on a *Retsch AS 200 Controle* analyses sieve. The following sieves were used for the feedstock 50, 25, 16, 12.5, 10, 8, 6.3, 4, 2.8, 1.4 mm whereas for the product 10, 8, 6.3, 4, 2.8, 2, 1.4, 1, 0.71, 0.5, 0.355, 0.25, 0.18, 0.125, 0.09, 0.071, 0.032 mm sieves were used. The amplitude of the sieve tower was kept at 1.5 mm for all analyses. In order to ensure reproducibility of the sieve analysis, the sieve time was determined for each sample according to DIN 66165-1 [**17**]. In addition, the comminution ratio $Z_{50,3}$ was calculated from the results of the sieve analysis. The definition of $Z_{50,3}$ is shown in Equation (2). Where $x_{50,3,\alpha}$ is the mean particle size of the raw material and $x_{50,3,\omega}$ the mean particle size of the product.

$$Z_{50,3} = \frac{x_{50,3,\alpha}}{x_{50,3,\omega}} \quad (2)$$

The moisture content of the materials was gravimetrically determined according to DIN EN 13183-1 [18]. The moisture content w of wood is defined, as shown in Equation (3). Therefore m_m is the mass of water in the sample and m_0 the mass of absolute dry wood.

$$w = \frac{w}{m_0+m_w} \quad (3)$$

Furthermore, the degree of dispersion was calculated according to VDI 3491 from the specific values of the volume size particle distribution [**19**]. The degree of dispersion characterizes the width of a volume size particle distribution. Depending on the width of the distribution particle collectives are termed as *monodisperse*, *quasi-monodisperse* or *polydisperse*. The degree of dispersion is defined according to VDI 3491, as shown in Equation (4). Therefore $x_{84,3}$, $x_{16,3}$ are characteristic particle size values where the volume size particle distribution is 84 % respectively 16 % and $x_{50,3}$ the mean particle size of the volume size particle distribution.

$$\xi = \frac{x_{84,3}-x_{16,3}}{2 \cdot x_{50,3}} \quad (4)$$

Materials

Three different types of fresh wood and two types of recycling material were chosen to investigate the influence on the comminution process and products as well as the energy requirement. The wood samples came from one deciduous and two coniferous species: common beech (*Fagus sylvatica L.*), common spruce (*Picea abies L.*) and oak (*Quercus robur L.*) as well as two types of quality of recycling wood: Quality type AI and AII. Recycling wood AI contains only pure wood, whereas recycling wood AII is a mixture of wood and fiberboard. The fresh wood was provided by the local wood retailer *Stabo Brennholz* in the form of whole logs. The recycling wood was provided by the recycling company *JRS J. Rettenmaier & Söhne.* **Fig. 2** shows the wood logs from the three different species and the recycling wood AI and AII.

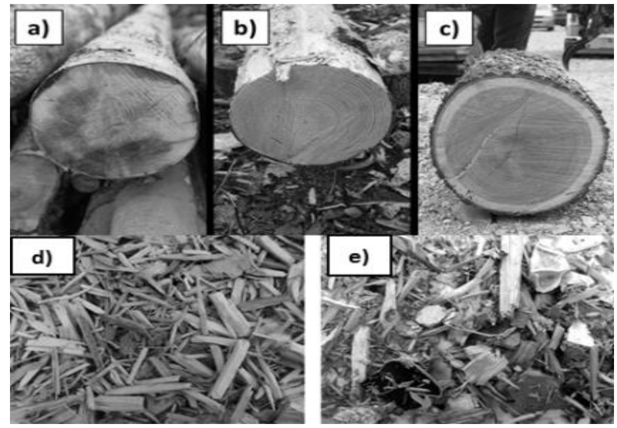

Fig. 2. Wood logs from three different species of fresh wood a) beech, b) common spruce and c) oak and the two types of quality, d) AI and e) AII, of recycling wood.

The wood chips, which constitute the raw material for further comminution experiments, were prepared from the logs (without the bark and preparatory drying) and recycling wood, using a chopper of the type *"Heizohack HM 8-500k"*. Around 200 kg of wood chips were produced from each species. The chopper provided a product which, according to DIN EN ISO 17225-4 [**20**], received the size class G30. DIN EN ISO 17225-4 is used to characterize biogenic solid fuels. Modern wood choppers produce wood chips according to one of the three size classes G30, G50 and G100. The size classes are an important particulate quality factor for the wood chip industry. **Table 1** shows the classification of mass fractions of wood chips according to the different size classes.

Table 1. Fractions of the Norm size G30 according to DIN EN ISO 17225-4.

Norm size	Main fraction	Fine fraction	Coarse fraction
G	> 60 %	< 20 %	< 20 %
G30	2.8 – 16 mm	< 2.8 mm	> 16 mm
G50	5.6 – 31.5 mm	< 5.6 mm	> 31.5 mm
G100	11.2 – 63 mm	< 11.2 mm	> 63 mm

At this state the moisture content of the beech was w_{beech} = 39%, common spruce w_{spruce} = 40%, oak w_{oak} =40% and the two types of recycling wood $w_{recycling}$ = 7%. The fresh wood chips were stored at room temperature until they reached the maximum moisture content for the experiments of w_{max}= 34 %. The maximum moisture content constitutes the moisture content from which the internal screens are not clogged by the material.

The recycling wood was comminuted at its initial moisture content of $w_{recycling}$ = 7%. The wood chips were split with a splitter into representative samples of 1.5 kg each. One-half of the samples were comminuted at the maximum moisture content and the other half was oven dried at a temperature of 105 °C to a minimum moisture content w_{min} = 1.5%. The raw materials were characterized after chipping by sieve analysis in terms of particle size distribution.

Results and discussion

Particle size analysis of the comminution products

Below, selected comminution results of beech, common spruce and oak wood chips with a moisture content of w = 1.5 % and w =34 % **(Fig. 3 and 4)** and the recycling wood AI and AII **(Fig. 5)** at a moisture content $w_{recycling}$ = 7 % are embodied. Due to a clearer arrangement and the identical trend of the particle size distribution of three reproducibility experiments, only one curve is displayed for each experiment. **Fig. 3** shows the cumulative particle size distributions of the beech, spruce and oak wood chips comminuted with the cutting and the hammer mill (in all figures hammer mill = HM and cutting mill = CM are abbreviated) at a moisture content of w = 34 %. In the cutting mill (continuous curve) internal screens of 4, 6, 8 and 10 mm and in the hammer (dashed curve) 4 mm mesh widths were used. Furthermore, the cumulative particle size distribution of the raw material is displayed (dotted curve).

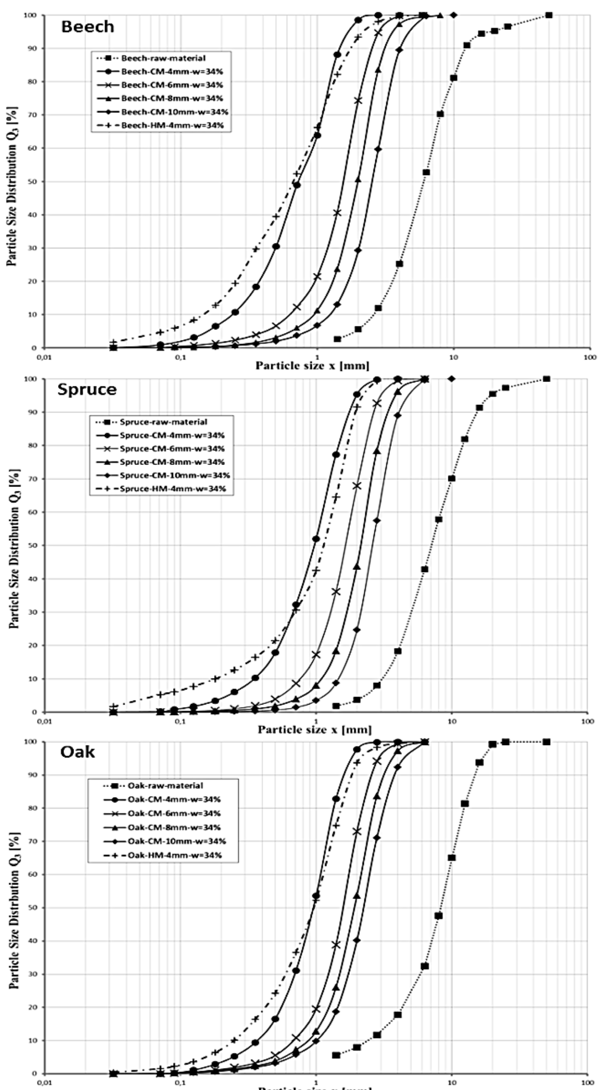

Fig. 3. Cumulative particle size distributions of the common spruce, beech and oak raw material and comminution products at a moisture content of w = 34 %

Fig. 4 shows the results of the experiments with the low moisture content (w= 1.5%) of the three different raw materials. Again the results from the cutting mill (continuous curve) with internal screens of 4, 6, 8 and 10 mm and in the hammer mill (dashed curve) of 4 mm are displayed as well as the cumulative particle size distributions of the raw materials (dotted curve).

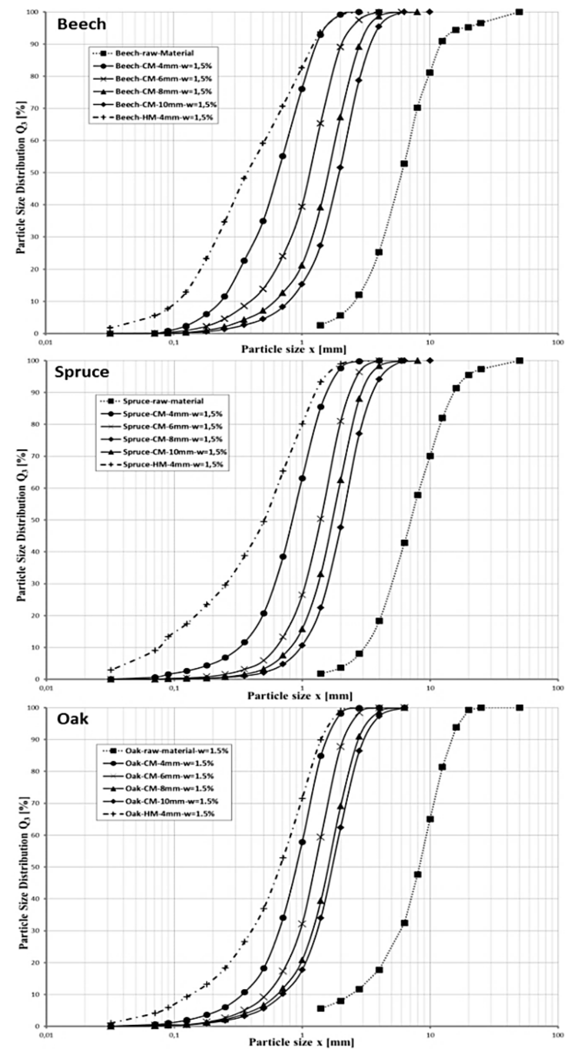

Fig. 4. Cumulative particle size distributions of the common beech, spruce and oak raw material and comminution products at a moisture content of w = 1.5 %.

In all cases of **Fig. 3** and **4** the comminution takes place over the entire size range and the curves of the cumulative particle size distributions shift towards finer particle sizes with the decrease of the mesh width of the internal screen. Comparing the results from the experiments with those of low moisture content of w = 1.5 % **(Fig. 3)** and the one with a moisture content of w = 34 % **(Fig. 4)** it can be concluded, that low moisture content leads to a finer product. If, for example, the mean diameters $x_{50,3}$ of the cumulative particle size distribution of the experiment with spruce in the cutting mill and internal 4 mm screen, is compared with each other, it is clearly visible that the product with low moisture content of w = 1.5 % and a mean diameter value of $x_{50,3} = 0.85$ mm **(Fig. 4)** is smaller compared to one with high moisture content of w = 34 % and a value of $x_{50,3} = 0.96$ mm **(Fig. 3)**. Comparable results can be obtained with all the experiments. The comminution product from the material with the lower moisture content is always finer than the one with the higher moisture content. This behavior is even more pronounced in the experiments with the hammer mill. It is most recognizable when comparing the experiments of spruce at the two moisture contents. The mean particle diameter of the product with lower moisture content with $x_{50,3} = 0.49$ mm **(Fig. 4)** is less than half the value of the one of high moisture content with $x_{50,3} = 1.12$ mm (**Fig. 3**). It is also clearly visible that the hammer mill produces a higher content of fine material when the wood is dry. Furthermore, the course of the cumulative particle size distributions from hammer and cutting mill differ strongly from each other. The slope of the cumulative particle size distribution of the products from the cutting mill is always steeper compared to the one of the hammer mill. That means the hammer mill produces particles with a wider volume density size distribution.

Fig. 5 shows the results of the experiments with two types of recycling wood at a moisture content ($w_{recycling} = 7$ %) of the two different raw materials. Again the results from the cutting mill (continuous curve) with internal screens of 4 and 10 mm and in the hammer mill (dashed curve) of 4 mm are displayed as well as the cumulative particle size distributions of the raw materials (dotted curve).

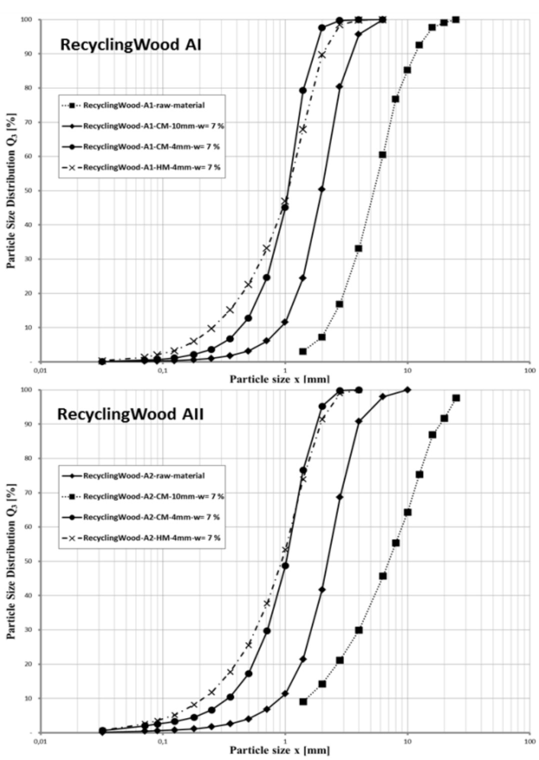

Fig. 5. Cumulative particle size distributions of the recycling wood (AI and AII) raw material and comminution products at a moisture content of w = 7 %.

In all cases of **Fig. 5** the comminution takes place over the entire size range and the curves of the cumulative particle size distributions shift towards finer particle sizes with the decrease of the mesh width of the internal screen. Again it can be obtained, that the comminution in the hammer mill leads to a product with higher content of fines. But for the experiments with recycling wood there is not difference between the mean diameter of the comminution from the cutting mill and the hammer mill. For the comminution products with the fresh wood **(Fig. 3 and 4)** there could be a big difference in the mean diameter, in dependency of the mill type, obtained. The slope of the cumulative particle size distribution of the products from the cutting mill is again always steeper compared to the one of the hammer mill. That means, once more, the hammer mill produces particles with a wider volume density size distribution.

Table 2 shows the comparison of the mean values of the degree of dispersion from the experiments with the cutting mill and hammer mill and an internal screen of 4 mm for the five different materials. Furthermore, the degree of dispersion of the raw materials is shown.

Table 2. Mean values of the degree of dispersion ξ from the comminution products of the hammer mill and cutting mill with an internal screen of 4 mm mesh width.

	ξ				
	Beech	**Oak**	**Spruce**	**Recycling Wood AI**	**Recycling Wood AII**
Raw Material	0.63	0.53	0.67	0.59	0.81
Product Hammer Mill					
w_{max}	0.92	0.71	0.66	0.69	0.75
w_{min}	1.23	0.77	1.01		
Product Cutting Mill					
w_{max}	0.71	0.50	0.59	0.47	0.57
w_{min}	0.69	0.51	0.55		

In **Table 2** the influence of the mill type, material and the moisture content can be seen. The moister content has a strong influence on the dispersity of the comminution product. For example, the degree of dispersion of the comminution product of beech with a moisture content of w =1.5% is with a value of $\xi = 1.23$ is nearly twice as high as the one from the product of the cutting mill with $\xi = 0.69$. This behavior could be also obtained with the other experiments. A degree of dispersion larger than $\xi > 0.41$ shows that the distribution is polydisperse. Furthermore, the product from the hammer mill has a wider volume density particle size distribution compared to the raw material, whereas the product from the cutting mill has a narrower volume density particle size distribution. Moreover, the achievable comminution ratio of the low moisture material is significantly higher in the hammer mill compared to the cutting mill. For example, with beech wood chips the comminution ratio in the hammer mill is with $Z_{50,3} = 16.46$ nearly twice as high as in the cutting mill with $Z_{50,3} = 9.02$. On the contrary, at high moisture content, the values of the achieved comminution ratio for both mills are similar. With the cutting mill the influence of the moisture content on the comminution ratio is by far not as distinct as with the hammer mill.

Specific energy requirement

Fig. 6 shows the specific comminution energy from three reproducibility experiments carried out with beech, spruce and oak at a high and low moisture content and the two different types of recycling wood plotted versus the degree of comminution $Z_{50,3}$. Results from the cutting mill are abbreviated with CM and from the hammer mill with HM.

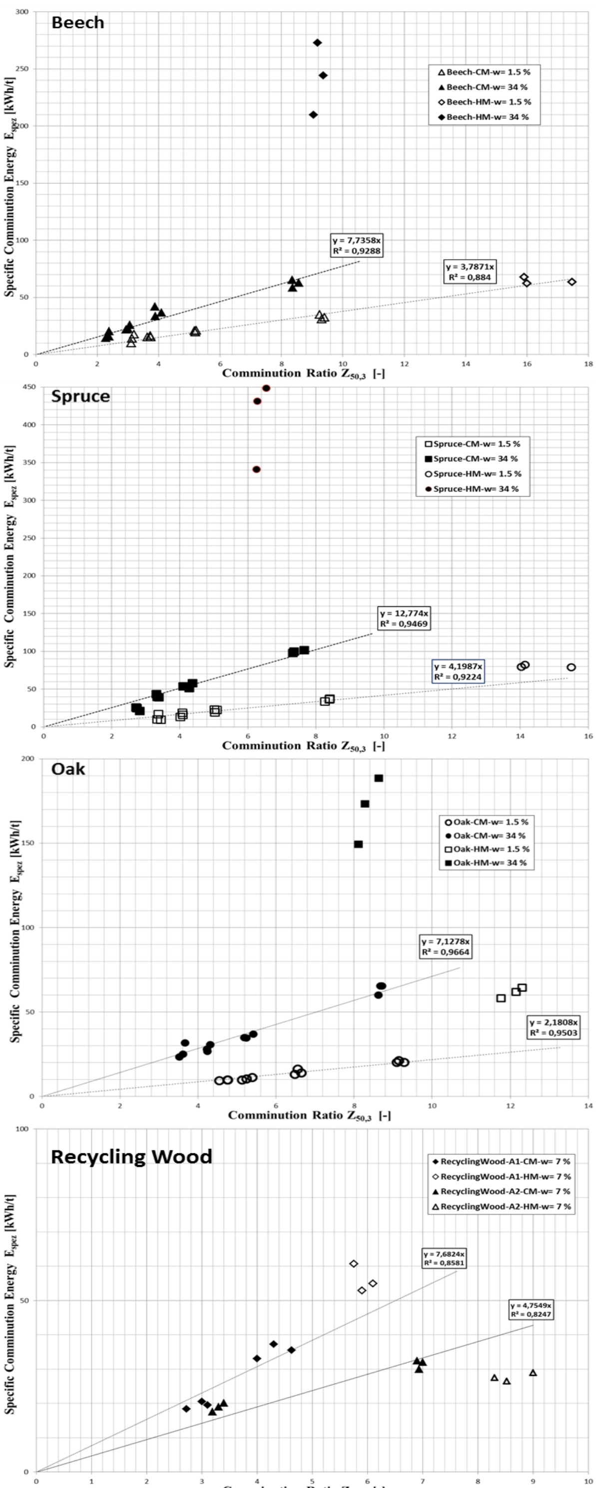

Fig. 6. Relationship between the specific comminution energy and the comminution ratio.

Fig. 6 comprises a linear correlation between the specific comminution energy and the comminution ratio with a high correlation coefficient of R^2 between 0.97 and 0.82 for all the materials and moisture contents when comminuted in the cutting mill. At the low moisture content of w = 1.5 % the gradient of the linear regression lines with all three materials is lower compared to the high moisture content w = 34 %. For both gradients of regression lines, the highest values exhibit spruce, whereas oak shows the lowest values. This behavior clearly indicates that the specific properties of the material, that determine their stress resistance, are changing individually with the moisture content according to the type of species. Furthermore, it is remarkable, that in **Fig. 6** the results from the experiments with spruce at the low moisture content are located on the same linear regression line for the hammer mill and the cutting mill. A similar result is obtained with the experiments with beech **(Fig. 6)** at low moisture content. The two materials, exhibit specific comminution energy at the low moisture content, which is not dependent on the type of stress but on the comminution ratio. This behavior is not observed in the experiments with oak. The results of the comminution of oak at the low moisture content with the hammer mill deviate strongly from the linear regression observed in the experiments with the cutting mill. At the high moisture content of w = 34 % for all materials the results from the hammer mill differ strongly from the linear regression line. In the hammer mill for all three materials, the comminution ratio decreases significantly at the high moisture content compared to the low moisture content, whereas the specific comminution energy increases dramatically. This strongly indicates that comminution of wood with the high moisture content is not advisable in hammer mill from the energetic scope.

For the comminution of the recycling wood there is also a linear relation between the specific comminution energy $E_{spez.}$ and the degree of comminution $Z_{50,3}$ with high correlation coefficient of R^2 between 0.86 and 0.82. The slope of the regression line for recycling wood AI is significantly higher compared to the one of recycling wood AII. That is because recycling wood AI mainly consist of pure wood particles and recycling wood AII is a mixture of pure wood and fiberboard. The mechanical strength of fiber board is much lower compared to pure wood which leads to the higher specific comminution energy of the recycling wood AI. The slope of the regression lines are in the same range like the ones of the other materials at low moisture content. Again, as for the comminution of oak, the results from the hammer mill have a strong deviation from the regression line of the experiments from the cutting mill.

Conclusion

In this work, the relationships between the specific comminution energy demand of wood chips, aperture size of the internal screens of the mill and the resulting particle size distribution were investigated. The experiments were carried out for beech, spruce, and oak at different moisture contents in a cutting and hammer mill. Furthermore two different qualities of recycling wood (AI and AII) where comminuted at their initial moister content w = 7 %. The moisture content of the biomass influences the comminution energy consumption significantly. At the high moisture content, the specific comminution energy of the materials was up to four times higher as compared to the low moisture content. This behavior is more distinct with softwood compared to hardwood. Furthermore, it is remarkable, that the observed relationships between the specific comminution energy and the moisture content for the fine comminution with cutting stress are in contradiction to the literature results for the coarse comminution (wood chipping) with the same type of stress. Bauerschlag *et al.* could show that for the chipping of wood the specific comminution energy decreases with an increase of the moisture content, whereas in this work it was clearly shown that the specific comminution energy increases with the moisture content [**21**]. The moisture content also influences the particle size distribution of the comminution product. Especially in the hammer mill, the lower moisture content, leads to a finer product with a higher content of fines and a wider particle size distribution. Also, the type of material shows a distinct influence on the specific comminution energy. Spruce exhibits the highest specific comminution energy at both moisture contents. The other two materials require lower specific comminution energy. The differences in the specific comminution energy of the three materials are more pronounced at the high moisture content. To what extent these relationships are caused by different structures of the materials and their different mechanical properties or due to the fact that the materials are different types of species - deciduous and coniferous wood – needs to be clarified in further studies. Regarding the type of mill, it can be concluded that the hammer mill is not appropriate for the comminution of the three materials with high moisture content in terms of energy consumption. However, the comminution ratio in the hammer mill at the low moisture content is remarkably higher compared to the cutting mill at the low moisture content. Furthermore, the products from the hammer mill have a wider volume density particle size distribution than the ones from the cutting mill. The relationship between the required specific comminution energy and the particle size distribution of the comminution product, which is one of the most important economic factors, show for all five materials a linear regression with different gradients. The specific comminution energy increases linearly with the increase of the comminution ratio. The gradient of these linear regressions is dependent on the moisture content as well as the type of material. These results lead to the assumption that these dependencies could be used to develop a new comminution model to predict the energy demand for the comminution of biogenic materials. Further studies have to confirm that. When commenuting recycled wood the specific comminution energy is dependent on the quality of the material. The higher the content of fiberboard in the material is the lower the specific comminution energy is.

Author's contributions

Conceived the plan: M. E., T. U.; Performed the expeirments: M. E; Data analysis: M. E.; Wrote the paper: M. E. Authors have no competing financial interests.

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