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Measuring methods and affecting factors - A review

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Review

Quality parameters relevant for densification of bio-materials: Measuring methods and affecting factors - A review

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ABSTRACT

Densification has been carried out for many years, mostly in biomass processing, animal feed production, and pharmaceutical industries. During the years, researchers and engineers attempted to improve the product quality and minimize the production costs. The most important quality parameters of solid bio-materials are the compressive strength, abrasion resistance, impact resistance, moisture adsorption, and density. Various studies used different standard and non-standard methods to characterize these quality parameters. The objective of this paper is twofold: (1) to investigate the state-of-the-art methods and devices used in the quality assessment of densified bio-materials, including a comparison between non-standard and standard methods. (2) to discuss the effect of different factors on the properties of densified bio-materials using an integrated approach. The results show a lack of standard methods for the quality assessment of bio-materials and therefore, there is an emerging need for development of dedicated standards for bio-materials. Moreover, the use of dissimilar methods and devices in the quality assessment of bio-materials gives risk to uncertainties about the effect of different factors on the product quality.

1. Introduction

Densification is the compacting process of material under specified conditions. Densification is classified into pelletization, briquetting, and extrusion [1]. According to Falk [2], the primary aim of pelletization is "the agglomeration of small particles into larger particles by means of a mechanical process in combination with moisture, heat, and pressure". Densification is widely used in biomass industries, animal feed making, and pharmaceutical industries. Generally, densification increases the bulk density, improves transportation and handling and logistics, decreases dust generation, and reduces the labor costs. Depending on the application, densification may provide also other advantages, for example, easy adaptation in direct-combustion or co-firing with coal, improving the flow properties of biomass, improving feed quality for animals and uniformity in mass and size of pharmaceutical products [3–9].

Pellet mills, hydraulic piston presses, mechanical piston presses, tabletizers, roller presses, and screw extruders are some examples of densification systems widely used in industry [10]. Densified materials are commonly cylindrical in shape, however, there are other shapes such as hexagons with or without a hole in the center. Although there are some standards for densified material size classification [11], there is no standard value to distinguish the pellets and briquettes by length and diameter size. According to CEN TS 14588 standard [12], the terms biofuel briquettes and biofuel pellets refer to densified biofuels made from pulverized biomass with or without pressing aids. The briquettes are cubic or cylindrical in shape, however, pellets are cylindrical with a typical random length of 5–30 mm with broken ends. Regarding the literature, most researchers used the term "Pellet" when the cylinder diameter was between 3 and 27 mm with a length of 3–31 mm [3–6,8,13–20] and some other authors used the term "Briquette" when the cylinder diameter was between 18 and 55 mm and the length was between 10 and 100 mm [21–29]. It is clear that for a diameter between 18 and 27 mm, both terms are used in the literature. In a study on wood

Abbreviations: B, Binder (molasses); BD, Bulk Density; D, Diameter; HT, Holding Time; HTC, Hydrothermal Carbonization; L, Length; MF, Mesocarp Fibre; MC, Moisture Content; NS, Not Specified; P, Pressure; PKS, Palm Kernel Shell; PD, Pellet Density; PSD, Particle Size Distribution; RD, Relaxed Density; T, Temperature; TD, True Density; TS, Tensile Strength; W, Water

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Table 1
Examples of national and international standards to measure quality parameters of different materials.

Standard Test	Quality parameter	Material
ASTM D2166–85:2008	Compressive strength	Wood
ASAE S 269.4	Density, durability, and moisture content	Cubes, pellets, and crumbles
CEN[a]/TS 15639:2010	Mechanical durability	Solid recovered fuels-Pellets
PFI[b]: Call Number: LD2668.T4 1962	Mechanical durability	Residential/commercial densified biomass
ISO 17831–1:2015	Mechanical durability	Solid Biofuels-Pellets
ISO 17831–2:2015	Mechanical durability	Solid biofuels-Briquettes
ÖNORM M 7135[c]	Mechanical durability	Wood pellets and briquettes
ASTM D 441-86	Mechanical durability	Standard test method of tumbler test for coal
ASTM D 440-86	Drop shatter test	Standard test method of drop shatter test for coal
DIN[d] 51705	Bulk Density	Solid fuels
EN 15103:2010	Bulk Density	Solid biofuels with a nominal top size of maximum 100 mm

[a] CEN: Common European Standard.
[b] PFI: Pellet Fuel Institute (USA).
[c] Austrian Standard.
[d] German Standard.

residue, densified cylinders of 49 mm in diameter and 50 mm in length are named as “Log” [1]. Other shapes of briquettes are also reported, for example, Chou et al. [30] made cubic briquettes of rice straw with the dimensions of 40 × 40 × 35 mm. In order to avoid confusion the terms pellets, briquettes, and logs in this paper are used in the same way as in the corresponding cited paper.

The suitability of the densification process is evaluated by measuring some of the physical properties of the final product. According to Richards [31], resistance to crushing, durability, impact resistance, and water adsorption are four crucial factors to be taken into account in developing and evaluating the densification process and quality of densified materials. He pointed out that there is a relationship between compressive strength, impact, and abrasion resistance. According to other researchers, density along with durability are the most significant factors in determining the physical quality of densified materials [8]. Czachor et al. [32] in their study of biomass pellets found that there is a relationship between density and physical quality of the pellets. Richards [31] stated that as the compressive strength increases, the density also increases, but the reverse is not always true since higher density does not necessarily stand for stronger bonding. Larsson and Samuelsson [33], showed that compressive strength of pellets highly depends on pellet density and durability where it can be modelled with a good fit.

Quality standards serve as a reference to provide customers with information about the quality and performance of products. Moreover, standards help to systematically assess the quality differences between the material of various origin and processes. It should be noted that the standards refer to firstly measuring methods such as using a standard tumbling device for durability measurement, and secondly, the product quality classification such as classification of pellets based on their durability.

By the advent of new densified bio-materials such as densified biomass and densified torrefied biomass in recent years, there is a concern about the performance of the existing transportation equipment in terminals and transportation units for large-scale transportation and storage. Research on biofuel demand in Northwest Europe carried out by Sikkema and Fiorese [34] shows that the import of woody biomass pellets for electricity generation may reach up to 16 Mt by 2035. The increasing biomass demand in other countries underlines the importance of transportation, handling, and storage. Presently, there are a few standards to measure the quality parameters of biomass-based materials, such as durability and density standards. In order to better understand the material behavior during transportation and storage, standards to measure the compressive strength, impact resistance, and moisture adsorption are required.

Several studies have reviewed different densification systems, their energy consumption, factors affecting densification processes, strategies to increase densified biomass durability, and bonding mechanisms [9,10,35,36]. However, there is no integrated approach that discusses the methods used to characterize the quality parameters of densified bio-materials. Based on an extensive literature study, the primary aim of this paper is to survey different quality assessment methods in detail and to investigate the effect of different experimental setups to the characterizations of material quality. This will be described in chapter 2 where the state-of-the-art experimental setups, their advantages and disadvantages, and comparisons with the existing standard methods are given. The other aim, which is outlined in chapter 3, is to investigate the effect of different factors on quality parameters of densified bio-material from integrated perspectives. Then, the results are discussed in chapter 4. The overall conclusions and future outlook will be outlined in chapter 5. It is worth mentioning that this paper is targeted to all the research and industrial units that are involved in bio-material production, handling and logistics, i.e. producers to end users.

2. Methods to measure the physical properties

Once the pellets or briquettes have been produced they are stored and transported to the end user location. During the transportation, the materials are subjected to several forces which may cause degradation [37]. The forces are divided into three main categories, namely compressive forces, shear forces, and impact forces [38]. Due to several limitations such as time, cost, unavailability of equipment, and on-site test difficulties it might not be possible to test the physical properties of the materials in the supply chain. Thus a number of tests, including compressive strength tests, impact tests, and abrasion tests were developed to simulate the conditions of the transportation, handling, and storage [9,22]. Despite the existence of standard methods for measurement of a selected number of physical properties shown in Table 1, there are many different methods in the literature to measure the strength of pellets or briquettes against these forces which are described in the following sections.

2.1. Compressive strength

Compressive strength measurements simulate the compressive forces acting on a sample during transportation and storage. For example, when the bulk material is transferred via belt conveyors or chutes or discharged into the storage silos, they encounter forces from either the equipment or the bulk material. Different devices have been used in literature to characterize the compressive strength [36,39]. The working principle of the majority of these devices is the same. The material is normally placed between two horizontal plates or a pressure piston and a bar which compress the sample at a constant rate until failure or breakage. Then the maximum force is recorded. Presently,

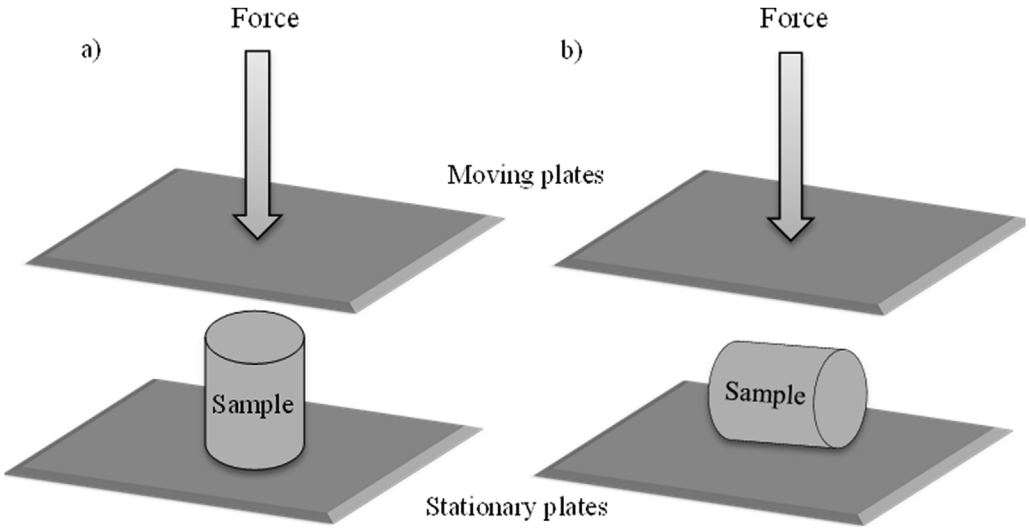

Fig. 1. Orientation of a typical crushing experimental setup during (a) compressive and (b) tensile strength testing adapted from Ref. [22].

there is no standard method for the compressive strength of densified bio-materials, however, according to the standard test method for compressive strength of cylindrical concrete specimens [40], a compressive axial load applies to the specimen until failure occurs. Then the compressive strength is calculated by dividing the maximum load by the cross-sectional area of the specimen. In literature, the compressive strength for densified bio-materials is defined as the maximum axial force (Fig. 1a) a material could withstand until failure or breakage or the maximum force during deformation [4,5,18,26,41]. If the force is applied perpendicular to the cylinder axis, it is called tensile strength (Fig. 1b). According to the standard test method for splitting tensile strength of cylindrical concrete specimens [42], in this test a diametrical compressive force applies along the length of the specimen until failure occurs. Then the tensile strength is calculated by:

$$T = \frac{2P}{\pi l d} \tag{1}$$

where T is the splitting tensile strength (MPa), P is the maximum load (N), l is the length (mm), and d is the diameter of the specimen (mm). However, some researchers do not follow these definitions and they use these terms conversely. Normally the compressive strength is higher than the tensile strength [22].

During the transportation and storage, the material encounters the forces from any direction. Therefore, some researchers argue that determining the tensile strength seems more practical than compressive strength because the tensile strength is related to the weakest orientation of the pellet or briquette [31]. Anyway, numerous researchers have only measured the compressive strength as an indication of the sample quality without giving any information about their choice argument [1,4,5,14,18,21,26,28,29,41,43].

Comparing the literature results enables to obtain a good understanding of the factors affecting the material properties. Nevertheless, variation in the test procedures and equipment in the literature mostly due to a lack of standard methods make it difficult or impossible to compare different material properties. For example, Kambo and Dutta [5] used a compression device to measure the strength of the pellets in the radial direction by applying a compression rate of 25 mm min^{-1} and reported the compressive strength as the maximum force that breaks the pellets. Hu et al. [4] also used the same procedure as Kambo and Dutta [5], but they applied a force rate of 2 mm min^{-1}. Yaman et al. [28] also used an Instron device to measure the compressive strength of fuel briquettes. They measured the compressive strength by dividing the maximum load to fracture the material over the cross-section area of the sample, however, the compression rate in their study was not stated.

Mitchual et al. [29] in their study of fuel briquettes used an Instron machine which compressed the cubic shape material at a rate of 0.305 mm min^{-1} and reported the compressive force using the equation (2):

$$\textit{Compressive strength}\ [N.\ m^{-1}] = \frac{3F}{l_1 + l_2 + l_3} \tag{2}$$

where F is the maximum force [N] crushing the material and l_1, l_2, and l_3 are the dimensions of briquettes [m].

Abdollahi et al. [18] and Svihus et al. [44] used a texture analyzer (Fig. 2, adapted from Ref. [45]) to measure the compressive strength of animal feed pellets. They placed the samples between a pressure piston and a bar horizontally and compressed the materials at the rate of 0.16 mm min^{-1} and recorded the maximum force at which the particle breaks. Then the compressive strength was reported as the maximum force in Newton.

Bergström et al. [14] investigated the compressive strength by positioning the samples between two parallel horizontal plates and compressed it in a radial direction at a rate of 0.4 mm min^{-1} until the sample was crushed. Then they reported the compressive strength according to equation (3):

$$\textit{Compressive strength}\ [N.\ mm^{-1}] = \frac{F}{L} \tag{3}$$

where L is the length of pellets [mm]. They were of the opinion that by dividing the force by the pellet length, the effect of length on the compressive strength was eliminated.

In the other study of densified solid fuels, Bazargan et al. [22] compressed the material perpendicular to the cylinder axis at a rate of 30 mm min^{-1} and measured the tensile strength using equation (4):

$$\textit{Tensile strength}\ (\sigma)\ [N.\ m^{-2}] = \frac{2F}{\pi D h} \tag{4}$$

where D and h are the pellet diameter and length, respectively. Liu et al. [46] also used the same procedure to measure the tensile strength of biomass pellets using a compression rate of 1 mm min^{-1}.

Chin and Siddiqui [25] have invented a test to measure the shear strength of biomass briquettes. They placed a sample on a 34 mm diameter and 12 mm deep stand and tied a piece of string of 5 mm diameter around the sample while the other end of the string was tied to a spring load using a pulley. Then the shear force which breaks the briquette was reported as the shear strength.

Richards [31] believes that using stress instead of compressive strength could remove the dependency of compressive strength on the

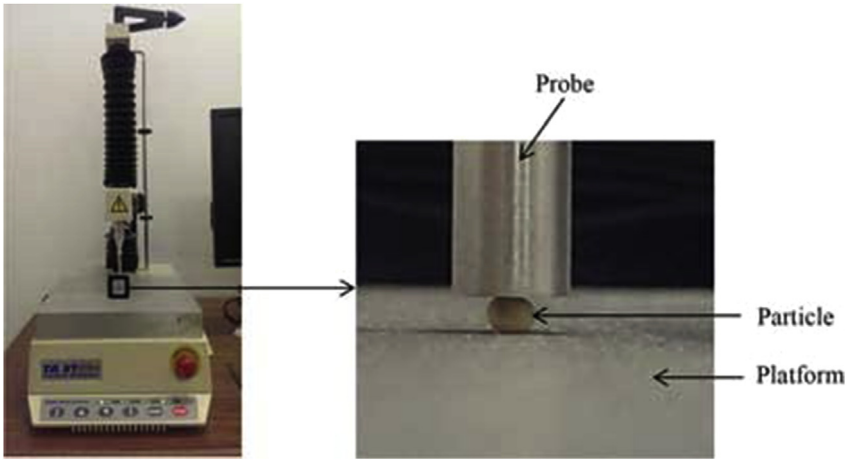

Fig. 2. A texture analyzer device adapted from Ref. [45].

shape and size of a briquette. The stress can be derived by equation (5).

$$Stress = \frac{load\ at\ fracture}{cross - sectional\ area\ of\ plane\ of\ fracture} \quad (5)$$

2.1.1. Meyer hardness

Another way of expressing the material strength is to determine the material hardness. Hardness is defined as the resistance to deformation. Brinell hardness, Vickers hardness, Rockwell hardness, and Meyer hardness are different kinds of hardness tests from which the Meyer hardness is most commonly used in literature to determine the hardness of densified bio-materials [3,15,47–51]. Meyer suggested the hardness should be based on the projected area of the impression rather than the surface area. Therefore, the Meyer hardness is the mean pressure between the surface of an indenter and the indentation i.e. the load divided by the projected area of the indentation [52]. Lam et al. [50] and Li et al. [15] believed that the Meyer hardness reflects the material strength during transportation and storage.

The Meyer hardness is measured by placing the sample between two anvils of a press while the force is diametrical. The maximum force a sample could withstand before breaking is measured and then the Meyer hardness is calculated from equation (6):

$$H_M = \frac{P}{\pi r^2} \quad (6)$$

where r is the indentation radius.

Tabil et al. [51] showed that the Meyer hardness could also be expressed by the indentation depth, thus the equation (6) could be expressed as equation (7):

$$H_M = \frac{P}{\pi\,(Dh - h^2)} \quad (7)$$

where D is the indenter diameter and h is the indentation depth.

Peng et al. [47] developed the equation (7) in order to determine the Meyer hardness for wood pellets. In their study, they indicated that as the surface of the wood pellets is mostly a curved shape, the cross-sectional area between the hemispherical probe and the pellet is oval-shaped. The developed equation is:

$$H_M = \frac{P}{\pi\sqrt{Dh - h^2}\sqrt{\frac{D_p^2}{4} - \left[\frac{\frac{D_p^2}{2} + \frac{D.D_p}{2} - D.h - D_p.h + h^2}{D + D_p - 2h}\right]^2}} \quad (8)$$

where D is the indenter diameter, h is the indentation depth before the pellet breakage, and D_p is the pellet diameter.

Peng et al. [49] used a 6.35 mm hemispherical probe on a press machine and compressed the samples positioned vertically at a speed of 1 mm min^{-1}, then used the above equation to characterize the Meyer hardness.

Regarding the probe shape and size and their effect on the Meyer hardness values, Tabil et al. [51] defined a number of experiments on different sizes of alfalfa cubes. Overall, they argued that the sphere-end shaped probe is more practical in Meyer hardness determination since firstly, the values obtained had a lower variance than a flat-end probe and secondly, it results in lower values of hardness corresponding to the occurrence of cracking in the cubes.

2.1.2. Bending strength

A bending test is used to determine the Young's bending modulus, i.e. the displacement of a material when different force values are exerted. By studying the bending test for rigid materials, the maximum force a material can withstand in bending can be determined. The principle of the bending test is similar to the compressive strength measurements, however, the force exerted on the material is concentrated on one spot. For instance, as shown in Fig. 3, the force is

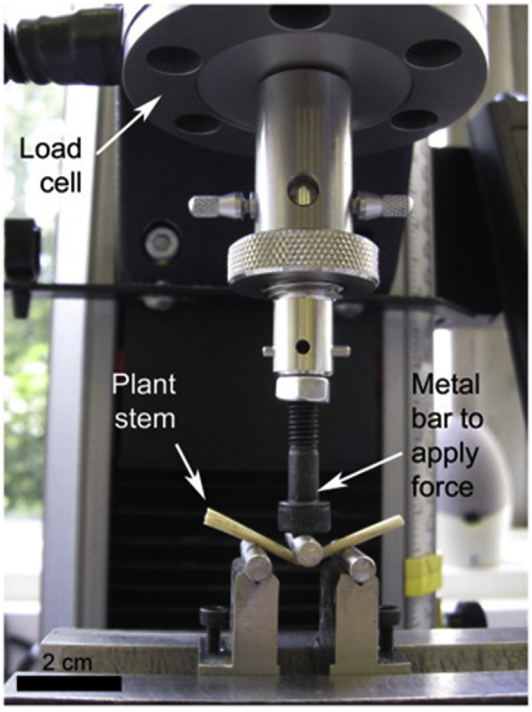

Fig. 3. Bending test on a sample of salt marsh canopies adapted from Ref. [53].

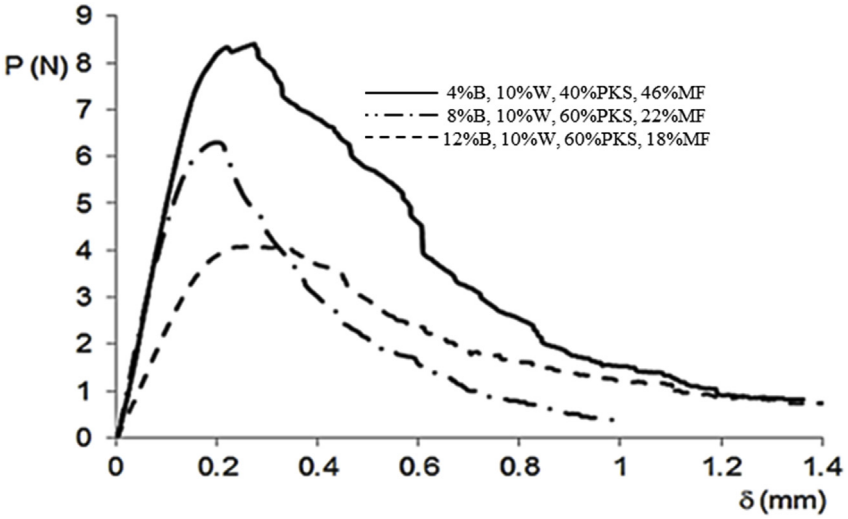

Fig. 4. Average curve of load vs deflection for biomass blend pellets adapted from Ref. [54].

orthogonally acting on the center of a plant stem.

Rupprecht et al. [53] measured the bending strength of different biomass plant stems using an Instron machine at a displacement rate of 10 mm min^{-1}. To measure the bending strength of palm oil biomass pellets, Arzola et al. [54] used a Shimadzu testing machine fitted with a 50 N load cell at 20 mm min^{-1}. Results of the later study are shown in Fig. 4.

2.1.3. Summary

Although different researchers used various rates of compressions in their studies, the effect of compression rates on the compressive strength or tensile strength is not clear yet and no study has been found to investigate this. Future research should be directed toward studying the effect of compression rate on the compressive and tensile strength in order to fill this gap. Furthermore, in order to reliably compare the Meyer hardness of different studies, the effect of geometrical factors such as indenter shape and size should be reported.

Some examples of compressive/tensile strength and Meyer hardness values of the above-mentioned literature are shown in Table 2 and Table 3, respectively. Although the compressive strength alone is not an indication of densified biomass strength during handling and transportation, Rahman et al. [55] believed that briquettes showing a compressive strength of at least 19.6 N mm^{-1} are suitable for handling for domestic purposes. Richards [31], suggested a minimum of 350 kPa (0.35 MPa) compressive strength is necessary for coal briquettes in order to withstand crushing in unclosed belt conveyors and normal bin storage. Nevertheless, the compressive strength test is widely used in characterizing the bio-materials strength, however, there is no dedicated standard procedure for densified bio-material. Therefore, there is an urgent need for the development of standard compressive test methods which encounters both pellets and briquettes in any shape and be capable of predicting the material breakage during transportation and handling.

2.2. Durability (abrasion resistance)

Presence of abrasive forces in the supply chain is highly likely. Hence, knowing the abrasion resistance is beneficial in order to decrease the risk of dust generation resulting in possible dust explosion, environmental risks, and waste generation.

According to standard terminology, definitions, and description of solid biofuels [12], mechanical durability is “the ability of densified biofuel units (e.g. briquettes, pellets) to remain intact during loading, unloading, feeding, and transport”.

Unlike the compressive strength which is tested using a single sample, the abrasion test is normally measured with multiple particles. Generally, a known mass of the screened pellets or briquettes is placed into the device which enables particle-particle and particle-wall interactions in a specified time. Then the amount of fines created is determined by means of sieving and finally, the durability is calculated based on the percentage of remaining mass on sieve divided by the initial mass. Different devices were used by researchers to determine the material durability such as the rotating drum, tumbling can, ligno tester, Holmen device, and electronic friabilator. The working principles and some examples of each test device are explained in the following.

2.2.1. Rotating drum

The rotating drum consists of a cylindrical chamber with baffles inside which rotates around its axial direction. A rotating drum of 101.6 mm in diameter and 95 mm in length was used by Reza et al. [16] to investigate the durability of torrefied pine pellets by using 10 pellets of the sample. Two baffles of 25.4 × 88.9 mm were installed perpendicular to the drum inner wall and opposite to each other and the drum rotated at 38 rpm for 3000 revolutions. After the revolutions, the sample was sieved through a 1.56 mm sieve size. In another study of mechanical properties of biomass pellets, Gil et al. [56] used a rotating drum of 130 mm diameter and 110 mm length, having two baffles of 30 × 110 mm perpendicular to the wall cell. They placed 40 pellets of 8 mm in diameter in the drum and rotated it for 3000 revolutions at 35 rpm. Then they used 2 mm mesh size sieve separate the created fine particles.

Temmerman et al. [57] used a rotating drum with a diameter and depth of 598 mm and a baffle of 598 × 200 mm perpendicular to the walls of the cylinder for measuring the durability of briquettes (Fig. 5). They used a rotational speed of 21 rpm and measured durability of different briquettes for different rotational times. Then used a 40 mm sieve size to separate the fine particles created at different drum rotation numbers.

2.2.2. Ligno tester

To characterize the pellets durability, Temmerman et al. [57] used a commercial ligno tester device according to the ÖNORM M 7135 [58]. As shown in Fig. 6, the device is a four-sided pyramid containing 2 mm round holes in each side. The particles are swirled by means of an air stream inside the equipment which causes the particles to collide with each other and against the walls. In their study, they used a standard air stream pressure of 70 mbar for one minute.

Table 2
Compressive or tensile strength of different densified materials.

Raw material		Shape and dimensions (mm)	PSD of raw material (mm)	Binder	Densification conditions	Density (Kg m^{-3})	Strength	Ref
Pyrolysed wood	@ 650 °C	Pellet D:20 L: 12-20	Mean size: 0.05–0.64	Alkaline lignin	P: 128 MPa	PD ~ 1100	~ 15 MPa	[4]
	@ 550 °C					PD ~ 1000	~ 9 MPa	
Miscanthus	HTC @ 190 °C	Pellet D: 6.35	< 0.73	No binder	P: 8.6 MPa HT: 10 s	PD: 887	~ 310 N	[5]
	HTC @ 225 °C					PD: 959	~ 275 N	
	HTC @ 260 °C					PD: 1036	~ 205 N	
	Torrefied @ 260 °C					PD: 820	~ 145 N	
Pine	Fine	Pellet D: 8	< 1	No binder	Pellet press (30 kW)	PD: 1263	61.2 N mm^{-1}	[14]
	Reference		< 8			PD: 1259	52.4 N mm^{-1}	
	Middle		1–2			PD: 1276	51.3 N mm^{-1}	
	Coarse		1–4			PD: 1274	40.1 N mm^{-1}	
Wheat based	Animal feed-starter period	Pellet D:3 L: 3	NS	Commercial pellet binder or moisture or no binder	Steam conditioning for 30 s T: 60 °C	NS	14.9 N with binder: 18 N with MC: 23.9 N with binder & MC: 23.4 N	[18]
					T: 90 °C		28.4 N with binder: 37.8 N	
	Animal feed-finisher period	Pellet D:3 L: 6			T: 60 °C		41.7 N with binder: 45.7 N 24.3 N with binder: 27.3 N with MC: 30.8 N with binder & MC: 29.4 N	
Gasified palm kernel shell		Briquette D: 25 L: 10-14	> 3 0.7–3 03–0.7 < 0.3	Starch & water	P: 80 MPa HT: 10 s	PD ~ 720	TS ~ 0.027 MPa TS ~ 0.022 MPa TS ~ 0.026 MPa TS ~ 0.035 MPa	[22]
Sawdust		Briquette D: 30	0.3–0.85	Molasses & starch	P: 10–100 bar	RD: 462	27.5–95.7 N	[25]
Coconut fiber			0.1–0.5			RD: 157	10–73.3 N	
Palm fiber			< 0.15			RD: 192	10–36.2 N	
Peanut shell			0.15–0.5			RD: 547	1.3–6.7 N	
Rice husk			0.1–0.18	water		NS	1.2–4.6 N	
Biomass-Lignite blends		Briquette D: 50 L: 100	< 0.25	Biomass	P: 250 MPa	NS	Without binder @ 40.7% MC: 1.1 MPa Without binder @ 10% MC: 11.8 MPa With binder @ 10% MC: 26.6 MPa	[28]
C. Pentandra		Briquette D: 55.3 L: 52.5	2–3.35	No binder	P: 50 MPa HT: 10 s	RD: 651	51.45 MPa	[29]
T. Scleroxylon						RD: 597	40.89 MPa	
A. Robusta						RD: 573	26.88 MPa	
T. Superba						RD: 673	24.67 MPa	
P. Africana						RD: 720	55.45 MPa	
C.Mildbreadii						RD: 655	19.18 MPa	
Maize cobs		Briquette D: 55.3 L: 52.5	< 1	No binder	P: 20–50 MPa HT: 10 s	RD: 541 to 659	0.12–0.54 N mm^{-1}	[43]
C. Pentandra						RD: 523 to 716	29.23–44.58 N mm^{-1}	
C. Pentandra: Maize cobs	90:10					RD: 565-742	27.29–59.22 N mm^{-1}	
	70:30					RD: 584-749	16.66–33.47 N mm^{-1}	
	50:50					RD: 588-774	7.72–24.04 N mm^{-1}	
Pinewood sawdust		Pellet D: 13.5 L.D^{-1}: 0.9	NS	No binder	P: max 280 MPa HT: 5 s	PD: 1141	3.91 MPa	[46]
Rice husk						PD: 1093	2.05 MPa	
Coconut fibre						PD: 984	1.51 MPa	
Coconut shell						PD: 1101	0.96 MPa	
Hydrochar of	Pinewood sawdust		< 0.15			PD: 1191	7.10 MPa	
	Rice husk					PD: 1334	4.21 MPa	
	Coconut fibre					PD: 1153	7.5 MPa	
	Coconut shell					PD: 411	2.97 MPa	

Bergström et al. [14] put 100 gram of pellets in a ligno tester device and rotated it twice for 30 s. The rotation velocity was not mentioned. Then the mass of abraded material was reported.

2.2.3. Holmen durability tester

The Holmen durability tester circulates the samples pneumatically inside the device by using an air stream in which particles collide with each other and with the equipment walls and creates fines. Normally, the test is conducted in less than two minutes. Abdollahi et al. [18] used a Holmen durability tester to measure the durability of animal feed pellets in 30 s sample circulation. Then the pellet durability index (PDI) was defined as the remained mass of samples on the sieve to the initial sample mass. The sieve size to separate the fines is not indicated in their study.

Table 3
Meyer hardness values of different densified materials.

Raw Material		Shape and dimensions (mm)	Binder	Densification Conditions	Pellet Density (kg m^{-3})	Meyer hardness (N mm^{-2})	Ref
Chinese fir		Pellet D: 7	Sewage sludge	P: 83 MPa HT: 30 s	863 with binder: 1160	3.02 with binder: 4.15	[3]
Camphor					883 with binder: 1144	2.87 with binder: 4.03	
Rice straw					1027 with binder: 1217	3.98 with binder: 4.18	
Torrefied Sawdust	@ 260 °C @ 270 °C @ 280 °C @ 290 °C @ 300 °C	Pellet D: 6.5 L: 12	Moisture	4000–6000 N HT: 30 s T: 70 °C	~1060 ~1050 ~1020 ~1010 ~1000	~3 ~3 ~4 ~4 ~3.5	[15]

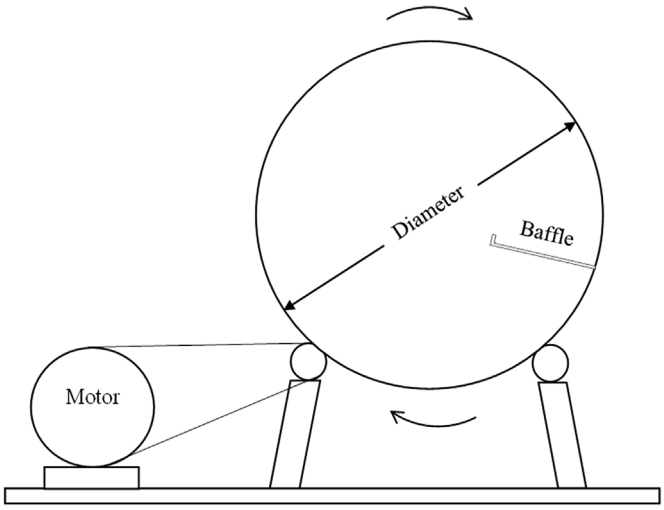

Fig. 5. Schematic of briquette durability tester adapted from Ref. [57].

2.2.4. Vibrating bed

Gilbert et al. [20] in the study of the durability of switch grass pellets, used a vibrating bed working at 5 Hz frequency and amplitude of 7–8 mm for 100 min and measured the mass loss of the original pellets. The equipment is shown in Fig. 7.

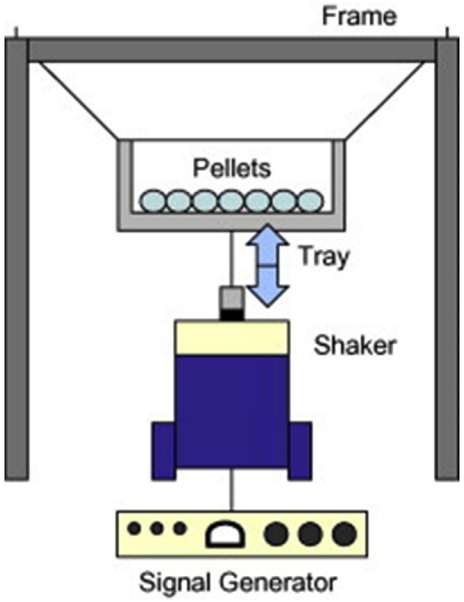

Fig. 7. Vibrating durability tester adapted from Ref. [20].

2.2.5. Tumbling can

Karunanithy et al. [59] and Fasina [60] used a commercial tumbling can durability tester device (Fig. 8) according to ASABE Standard

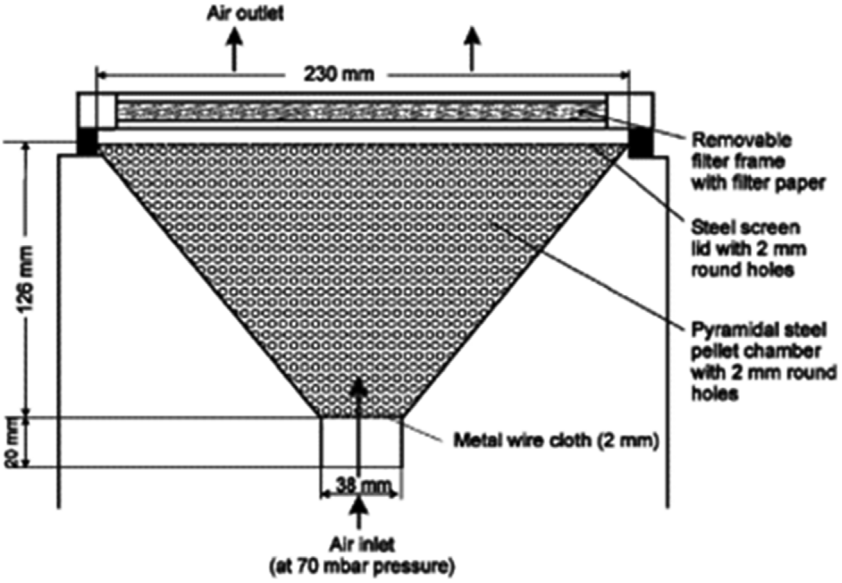

Fig. 6. ÖNORM M 7135 apparatus for durability testing of pellets [57].

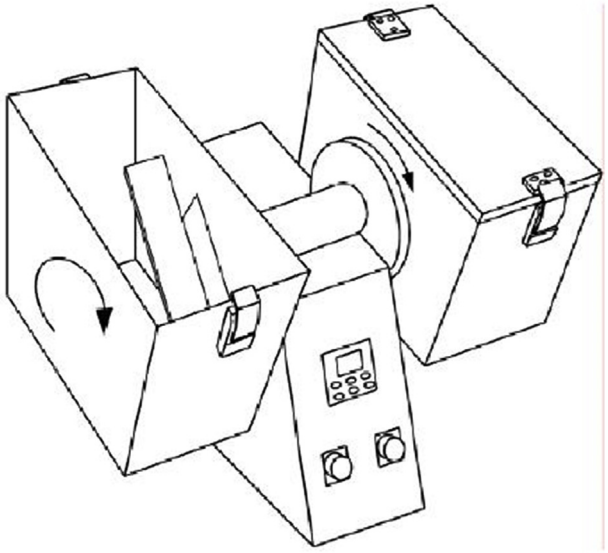

Fig. 8. Schematic of a tumbling can tester adapted from Ref. [61].

S269.4 [61]. They placed 100 g of samples into the device and rotated it at 50 rpm for 10 min and used the sieve sizes of 4.75 mm and 4 mm to separate fines, respectively. They reported the durability as the mass of particles remaining in the sieve to the initial mass of material.

2.2.6. Friabilator

Friabilators form another class of durability testers. Friabilators are mostly used in the pharmaceutical industries in order to measure the durability of pharmaceutical products. A typical friabilator contains a cylinder low in depth (compared to the diameter) with a curved baffle attached between two walls of the cylinder (Fig. 9). Zainuddin et al. [6] used a commercial friabilator to examine friability of animal feed pellets. They placed 20 pellets into the drum and rotated this for 4 min at 25 rpm. After 100 rotations they measured the fines created and reported the friability by dividing the mass of fines created to the initial mass of pellets.

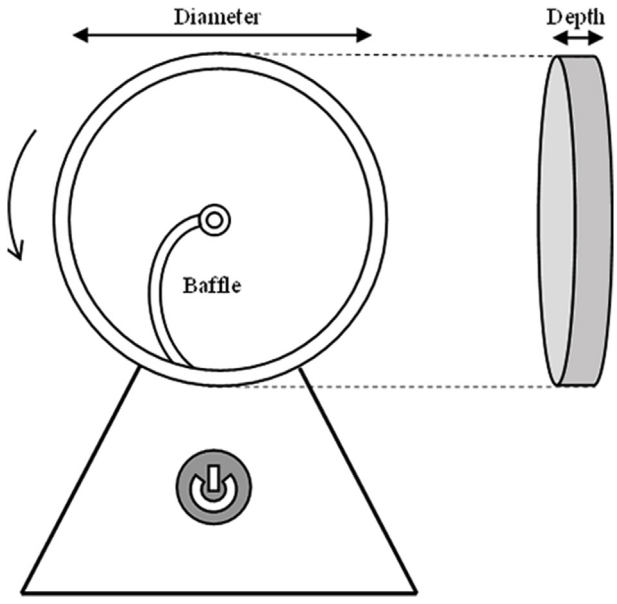

Fig. 9. A typical friabilator.

2.2.7. Other durability testing methods

Li and Liu [1] used a porcelain jar to measure the abrasion resistance of logs made from wood residues. Three logs of 49 mm in diameter and 50 mm in length were put in the jar and were rotated at 60 rpm for 40 min. The mass loss of the logs indicated the durability.

Sengar et al. [23] constructed a cuboid steel box with the dimensions of 30 × 30 × 45 cm and used a hollow shaft to set the frame diagonally, then rotated it for 15 min. They did not mention the other details of their set up such as the rotational frequency. They measured the durability index using equation (9):

$$Durability\ index = 100 - \%\ mass\ loss\ of\ the\ initial\ sample \quad (9)$$

Umar et al. [19] used an Erlenmeyer flask to measure durability of animal feed pellets. They placed 100 g of pellets sieved on a 2.36 mm mesh size into an Erlenmeyer flask and tumbled on a shaker at 50 rpm for 10 min. Then they used the aforementioned sieve to separate the fines and reported the pellet durability index (PDI) according to EN15210-1 [62] as follows:

$$PDI = \frac{Pellet\ mass\ after\ second\ sieving}{Pellet\ mass\ before\ tumbling} \times 100 \quad (10)$$

Schulze [63] has explained an attrition test procedure using a ring shear test. The ring shear test is a device mostly used to measure the angle of internal friction. In a ring shear test apparatus, the material fills the shear cell and the shear cell is placed on the tester and covered by a lid. Then the sample is sheared to a pre-determined shear displacement. Shear displacement is the relative rotational displacement of the bottom ring and lid which is measured at the mean radius of the sample. The amount of fines created by the test determines the vulnerability of samples.

2.2.8. Summary

Regardless of the test device type, the chosen sample volume in each test batch in the literature was less than 2% of the drum volume. None of the authors pointed out how the sample volume was chosen. It is clear that the more free space inside the drum allows particles to freely move and interact with other particles and equipment wall, and vice versa.

Considering the equipment type and sieve size used, even the standard methods are modified by some changes by the researchers. For example, to use the standard tumbling method, Reza et al. [14] used a 1.56 mm sieve size and Temmerman et al. [57] used a 40 mm sieve size while according to the standard the sieve size should be 3.15 mm. Therefore, it should be noted that the reported durability values in the literature are not comparable unless a complete standard method was used. Consequently, not all the high durability values in the literature are acceptable from the standard or practical point of view.

Some examples of the durability values of various pellets and briquettes using different raw material and test methods are shown in Table 4. Besides the differences between the devices and device setup, there are still differences between the experiments. As an example, Lindley and Vossoughi [26] measured the durability of a single briquette by a tumbling can method while most of the researchers used a batch of particles in their durability test devices. It should be highlighted that when using a single briquette in tumbling can, the method misses the abrasion forces between samples and may increase abrasion between briquette and equipment wall which may lead to different values of durability compared to the use of a group of particles. Another approach followed is that several researchers have reported the durability based on the amount of releasing fines while some other researchers reported that based on the mass loss of the initial sample. It appears that the former is mostly used for pellets while the latter is mostly used for briquettes. However, neither durability values nor dust generation characteristics are comparable when using these two different methods.

Table 4
Durability of different densified materials.

Raw Material		Shape and dimensions (mm)		PSD of raw material (mm)	Binder	Densification Conditions		Density (kg m^{-3})	Durability (%)	Ref
Oak		Log D: 49 L: 50		< 50	No binder	P: 34–138 MPa HT: 0–60 s		Max: 915	@ P 138 MPa: 98.3	[1]
Oak bran								Max: 1006	@ P 138 MPa: 97.6	
Pine								Max: 980	@ P 138 MPa: 93.2	
Cottonwood								Max: 960	@ P 138 MPa: 98.5	
Animal feed (pineapple)		Pellet D: 8 L: 30		< 1	35% Moisture	Extruder T: 100 °C		BD: 303 TD: 1520	99.15	[6]
					40% Moisture			BD: 323 TD: 1514	99.09	
					45% Moisture			BD: 323 TD: 1508	98.98	
					50% Moisture			BD: 345 TD: 1503	98.78	
Pine	Fine	Pellet D: 8		< 1	No binder	Pellet press (30 kW)		PD: 1263	98.8	[14]
	Reference			< 8				PD: 1259	99.1	
	Middle			1 to 2				PD: 1276	99.1	
	Coarse			1 to 4				PD: 1274	98.4	
Torrefied loblolly pine @	250 °C	Pellet D: 13 L.D^{-1}: 0.6 to 0.75		0.6 to 1.18	Moisture	P: 250 MPa HT: 30 s		PD: 1048	77.3	[16]
	275 °C							PD: 1012	78	
	300 °C							PD: 931	55.6	
	350 °C							PD: 689	9.3	
	250 °C				10 wt.% HTC loblolly pine 260 °C			PD ~ 1110	95	
	275 °C							PD ~ 1010	92	
	300 °C							PD ~ 950	83	
	250 °C				50 wt.% HTC loblolly pine 260 °C			PD ~ 1120	98	
	275 °C							PD ~ 1080	99	
	300 °C							PD ~ 1050	97	
	350 °C							PD ~ 730	92	
loblolly pine HTC@	260 °C				No binder			PD ~ 1430	99.8	
Wheat based	Animal feed-starter period	Pellet D: 3 L: 3		NS	Commercial pellet binder & moisture	Steam conditioning for 30 s	T: 60 °C	NS	56.5 with binder: 63.1 with MC: 67.2 with binder & MC: 70.2	[18]
							T: 90 °C		63.2 with binder: 69.6	
	Animal feed-finisher period	Pellet D: 3 L: 6							92.8 with binder: 93.1	
							T: 60 °C		74.1 with binder: 73.9 with MC: 84.7 with binder& MC: 89.7	
Malaysian mahseer		Pellet D: 3		NS	Tapioca-sago starch	P: 8–10 MPa		BD: 421 to 491	81 to 86.6	[19]
Switch grass		Pellet D: 26.8 L: 20 to 31		10 to 70	No binder	P: 4.1 MPa P: 55.2 MPa	HT: 30 s	PD: 310 to 505	95 98.5	[20]
Cashew nut: Grass: Rice husk	50:25:25	Briquette	D: 22.5 L: 60.5	NS	No binder	NS		BD: 895	95	[23]
	25:50:25		D: 22.7 L: 53					BD: 1105	93	
	25:25:50		D: 22.4 L: 49.8					BD: 1109	92	
Pine sawdust		Pellet D: 8		< 1	–	A commercial tablet press was used		–	~88	[56]
Chestnut sawdust									~93	
Eucalyptus sawdust									~36	
Cellulose residue									~70	
Coffee husks									~10	
Grape waste									~2	
Bituminous coal				< 0.212					~75	
Anthracite coal									~0	
Mixed wood		Briquette		–	–	Commercial briquettes		–	@ 105 rotation ~95	[57]

(*continued on next page*)

Table 4 (*continued*)

Raw Material	Shape and dimensions (mm)		PSD of raw material (mm)	Binder	Densification Conditions	Density (kg m^{-3})	Durability (%)	Ref
							@ 210 rotation ~90 @ 315 rotation ~84 @ 420 rotation ~78 @ 630 rotation ~68	
Mixed wood	Pellet	D: 6-8	–	–	Commercial pellets	–	99 to 99	
Softwood		D: 6-8					91 to 99	
Hardwood		D: 6					91	
Straw		D: 9-10					93 to 98	
Corn stover Switchgrass Prairie cord grass Sawdust Pigeon pea grass Cotton stalk	Briquette D: 60		< 3.36	No binder	A commercial briquetting machine was used	–	~90 ~78 ~72 ~89 ~55 ~88	[59]
Peanut hull	Pellet D: 4.76		< 3.18	Steam added	T: 88 °C Pellet mill was used	PD @ 9.1% MC: 1422 BD @ 9.1% MC: 634	90.3	[60]

2.3. Impact resistance

The impact resistance which is also called as shattering resistance or shattering test or drop test measures the resistance of samples when dropping from a known height onto a known floor material. Kambo and Dutta [5] believed that by measuring the impact resistance it is possible to investigate the forces acting on pellets when unloading from trucks to the ground surface or transferring the material from chutes into bins and also resistance during pneumatic conveying. Although many tests have been set to measure the resistance of the densified material against shattering, there is no standard method for densified biomass [64]. Mostly, researchers design drop test experiments based on their knowledge or imitate the other literature. Meanwhile, some researchers used another material's standard (e.g. coal and concrete) tests in their experiments.

Richards [31] has introduced the impact resistance index (IRI) for the fuel briquettes based on the drop number and number of pieces created. He dropped single briquettes for 3 to 6 times from a height of 2 m onto a concrete floor until the briquettes broke down into smaller pieces. Then he recorded the average number of pieces created and defined the IRI as below:

$$IRI = \frac{Average\ number\ of\ drops}{Average\ number\ of\ pieces} \times 100 \quad (11)$$

He proposed the minimum acceptable value for IRI of a laboratory work is 50.

Mitchual et al. [43] used the ASTM standard D440 which is a test method of drop test for mineral coal and Li and Liu [1] adapted their drop test from that standard test. The test procedure is to drop the material from a 2 m height onto a concrete floor and measuring the resistance index using the equation (11) while only the created pieces of bigger than 5% of the initial mass of material are taken into account. Demirbas and Sahin-Demirbas [65] used the standard method of coke shattering indices (ISO 616:1995). The test consists of dropping the material from the height of 1.8 m onto a steel plate, then the drop resistance is measured by determining the portion of material retained on a sieve having a 20 mm mesh size. This is repeated until all the material pass the aforementioned sieve. The sum of percentages is called shatter index [28].

Sengar et al. [23] dropped the briquettes from a height of one meter onto a RCC floor and concrete floor and reported the shattering resistance by the following equations:

$$Shattering\ resistance = 100 - \%\ mass\ loss \quad (12)$$

$$\%\ mass\ loss = \left(\frac{Initial\ mass\ of\ briquettes - Final\ mass\ of\ briquettes}{Initial\ mass\ of\ briquettes}\right) \times 100 \quad (13)$$

Al-Widyan et al. [27] and Kambo and Dutta [5] measured the durability of olive cake briquettes by dropping them four times from a height of 1.85 m on a steel plate and measured the durability as the final mass retained on the briquette after falling.

Oveisi et al. [66] placed 100–5000 g biomass pellets in an enclosed bag made of synthetic material and released the bag from different heights onto a concrete floor. Weatherstone et al. [64] collected the material in bags of 300 and 2000 g after which they dropped them from a height of 7.52 m for 10 times. Then, the impact resistance was reported based on the particles bigger than 3.15 mm and 3.16 mm, respectively. Moreover, the former research studied the effect of sample cushioning and concluded that by increasing the sample mass from 1000 to 5000 g, the increase in mass loss is smaller. Nonetheless, the effect of the bag cushioning was never studied.

2.3.1. Rotary impact device

Wu et al. [67] used a rotary impact test device (Fig. 10) to measure the particle breakage. The material was fed between two parallel discs which rotated with a pre-determined speed. The material shoots out and hits the steel plates inside the apparatus. The fines created during the test were determined. They used two different disc tip speeds of 6.5 and 24.3 m s^{-1} in a tangential direction in order to simulate higher limit of impact in practice and then they measured the created fines by sieving the material by two sieve sizes of 2.8 and 6.3 mm. The results are showing in Table 5. As expected, the amount of fines created for 24.3 m s^{-1} disc tip speed tests is higher than for the tests performed at

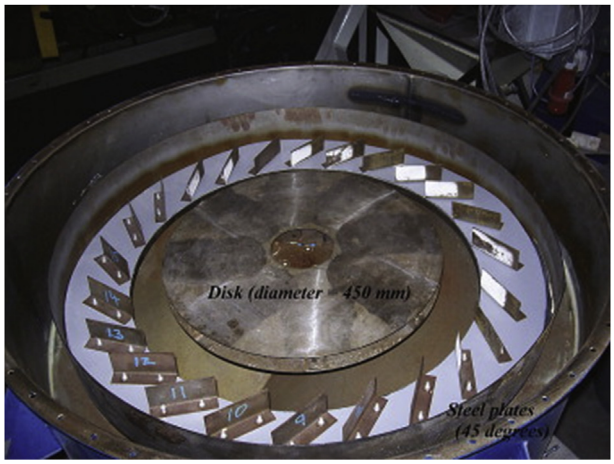

Fig. 10. Rotary impact test apparatus of the University of Greenwich used by Wu et al. [67].

6.5 m s^{-1}.

2.3.2. Summary

The impact resistance values of different materials are shown in Table 5. No research has been found in the literature to discuss the kinetic energy of samples and the impact force value at the impact point. However, these values depend on many factors such as sample mass, sample shape, and velocity at the impact point.

To figure out the material strength during transportation, Richards [31] believed that there is a relationship between the compressive strength, impact resistance index and abrasion resistance. He showed that briquettes with compressive strength values of higher than 375 kPa and IRI of higher than 50, usually show more than 95% abrasion resistance. Therefore, he suggested a drop test could be used as a guideline to estimate the strength of the material before conducting compression or durability tests. If the minimum acceptable quality is reached, then the other tests such as the compressive strength test or abrasion test could be investigated. Nevertheless, the test seems to be the simplest test method for evaluating the material strength in terms of facilities, laboratory work, time, and cost. However, similar to the compressive strength, lack of a standard test method has resulted in widely differing results in the literature which makes them incomparable.

2.4. Density measurements

Density can be expressed in three different ways namely granular density, particle density, and bulk density. The granular density (or true density) is the density of the material without porosity, the particle density is the density of densified material (like pellets or briquettes) considering the inner porosity, and finally, the bulk density is the density of a group of material containing the porosity between particles.

Different materials show different ability to compress. Therefore, "degree of densification" showing the ability of the material to bond has been defined [68] as shown in equation (14).

$$\text{Degree of densification} = \left(\frac{\text{Particle density of densified material} - \text{Density of raw material}}{\text{Density of raw material}}\right) \times 100 \quad (14)$$

When measuring the particle and bulk densities, one should pay a serious attention to the volume expansion or shrinkage of the material which might occur immediately after densification in both axial and

Table 5
Impact resistance of different densified materials.

Raw Material		Shape and dimensions (mm)		PSD of raw material (mm)	Binder	Densification Conditions	Density (kg.m^{-3})	Impact resistance (%)	Ref
Olive cake		Briquette D: 25		NS	35% Moisture	P: 15–45 MPa	RD: 1100-1300	75–99.25	[27]
Cashew nut: Grass: Rice husk	50:25:25	Briquette	D: 22.5 L: 60.5	NS	No binder	NS	BD: 895	97	[23]
	25:50:25		D: 22.7 L: 53				BD: 1105	95	
	25:25:50		D: 22.4 L: 49.8				BD: 1109	94	
Miscanthus	HTC @ 190 °C	Pellet D: 6.35		< 0.73	No binder	P: 8.6 MPa HT: 10 s	PD: 887	~92.5	[5]
	HTC @ 225 °C						PD: 959	~94	
	HTC @ 260 °C						PD: 1036	88.8	
	Torrefied @ 260 °C						PD: 820	~83	
Rice husk & Corn cobs blends		Briquette D: 32 L: 100		Rice < 2 Corn < 1.6	Starch	P: 19–31 MPa	NS	90	[21]
Flax Straw		Briquette		< 6	Moisture	P: 35.2–91.4 kg cm^{-2}	PD: 1069	97.1	[26]
Wheat Straw		D: 18		< 25		P: 58.4–84.4 kg cm^{-2}	PD: 1056	98.8	
Sunflower		L: 50				P: 31.6–98.4 kg cm^{-2}	PD: 1432	99.2	
Torrefied pellets		Pellet D: 6		NS	NS	Commercial pellets	@ 6.5 m s^{-1}	S11: 99.9 S22: 99.9	@ 24.3 m s^{-1} S1: 97.8 S2: 93	[67]
Wood pellets		Pellet D: 6						S1: 99.8 S2: 95.8	S1: 95.9 S2: 84.1	
		Pellet D: 8						S1: 99.7 S2: 99.3	S1: 97.4 S2: 94.9	
		Pellet D: 12						S1: 99.5 S2: 98.8	S1: 96.2 S2: 91.1	

[1] S1: 2.8 mm sieve.
[2] S2: 6.3 mm sieve.

lateral directions [3,8,20]. Carone et al. [8] reported up to 5% expansion in diameter of pellets having an original diameter of 6 mm and Gilbert et al. [20] observed around 10% decrease in pellet density of switchgrass one hour after densification. According to Jiang et al. [3], the volume expansion, on the one hand, creates more pores inside the densified material causing less resistance against abrasion and compression forces, and on the other hand, may produce a remarkable amount of fines before any transportation or handling activity. Moreover, the expansion creates more fines at the surface of the material causing coarser surfaces which might inflict more fine production in the future handling compared to smooth surfaces. Al-Widyan et al. [27] observed more than 10% shrinkage in the axial direction of briquettes made from olive cakes. They believed that the reason lies in the excessive loss of moisture content from the briquettes after densification.

In the followings, different methods to determine the granular, particle, and bulk densities are discussed.

2.4.1. Granular density

The granular or true density is mostly measured using a "Pycnometer". The measurement is according to the pressure difference between a pre-determined reference volume and the sample cell volume. A schematic of a typical gas displacement pycnometer is shown in Fig. 11. A sample of a known mass is placed into the volume calibrated sample cell. First, valve 1 opens to flow the inert gas into the chamber. Then valve 1 closes and one lets the chamber to reach equilibrium conditions. Once the equilibrium is reached, the pressure value is recorded. After that, valve 2 opens to allow the gas to go through the reference cell. After the whole system reaches an equilibrium condition the pressure is recorded again. Finally, the solid sample volume is calculated based on the pressure difference between the first and second equilibrium conditions.

Zainuddin et al. [6], Karunanithy et al. [59], and Fasina [60] used helium gas to fill the reference and sample cells. The true density was then measured based on the following equation:

$$True\ density = \frac{m}{V_{cell} - \frac{V_{exp}}{\left[\left(\frac{P_1}{P_2}\right) - 1\right]}} \quad (15)$$

where m is the sample mass, V_{cell} is the empty volume of the sample cell, V_{exp} is the expansion volume, P_1 is the pressure before expansion, and P_2 is the pressure after expansion.

2.4.2. Particle density

Particle density also known as apparent density, intrinsic density [22], or relaxed density [27]- if measured after a certain time from the material's production-is the ratio of mass and sample volume including pore volume [57]. Normally, densified materials are not smooth in shape which creates measurement difficulties in practice. Therefore, several studies tried to measure the volume by applying different methods. Temmerman et al. [57] used the buoyancy method (based on the Archimedes principle) to estimate the volume of different pellets and briquettes. The sample mass is measured in air and in a liquid with a known density. Then the volume of the sample can be calculated based on the liquid density. The method might create difficulties in practice since some material disintegrate in the liquid, quickly. The other disadvantage of the method is that the liquid might go inside the pores resulting in errors in the experiments then it may rather determine the true density.

Another method is to immerse the particles in a liquid while coated with wax. Sengar et al. [23] used this method for the volume determination of biomass briquettes. First, they coated each briquette with wax and then weighted it. Secondly, they immersed the coated sample in water and the displacement water was measured indicating the wax briquette volume. Then the volume of each briquette was calculated by the difference between the coated briquette volume and coating wax volume. The volume of coating wax was obtained by dividing its mass by its density. The mass of the coating wax was also determined by subtracting the mass of wax briquette and the original briquette. Comparing the previous method, this method prevents water adsorption inside the material pores.

Several studies used an easier method to determine the particle density [5,29,67,69]. They measured the mass of each pellet or briquette by using a laboratory balance and measured the material volume based on the diameter and length of the sample measured by a caliper. Then the ratio of mass to the volume was determined as the particle density. The advantage of this method is that a rough estimate of the particle density is achieved very quickly and simply, however, the disadvantage is that the method is not precise because the volume is not accurately determined. Some researchers have modified this method in order to improve the results. For example, Mitchual et al. [29] measured the diameter and length of cylindrical briquettes at three different points and calculated the particle density ($g.cm^{-3}$) using the average value of the diameter (mm) and length (mm) according to equation (16).

$$Particle\ density = \frac{108000 \times mass\ of\ particle}{\pi \times (d_1 + d_2 + d_3)^2 \times (l_1 + l_2 + l_3)} \quad (16)$$

2.4.3. Bulk density

Bulk density is the mass ratio of a known volume of the bulk material to its volume including the voids between particles. The mass and volume are measured using a balance and a container with a known volume, respectively. The European bulk density measurement standard for solid biofuels EN 15103 [70], states that the volume of the container could be between 1 and 50 liters depending on the solid biofuels size and the quantity available. Karunanithy et al. [59] used the ASAE standard method to measure the bulk density of ground feedstocks and briquettes. They used a 2000 ml glass container and calculated the bulk density by dividing the mass of the material to the glass volume. Zainuddin et al. [6] filled a 200 ml cylinder with the pellets of 8 mm diameter and 3 cm length and tapped the container twice to obtain a uniform packing and reduce the wall effects. Wu et al.

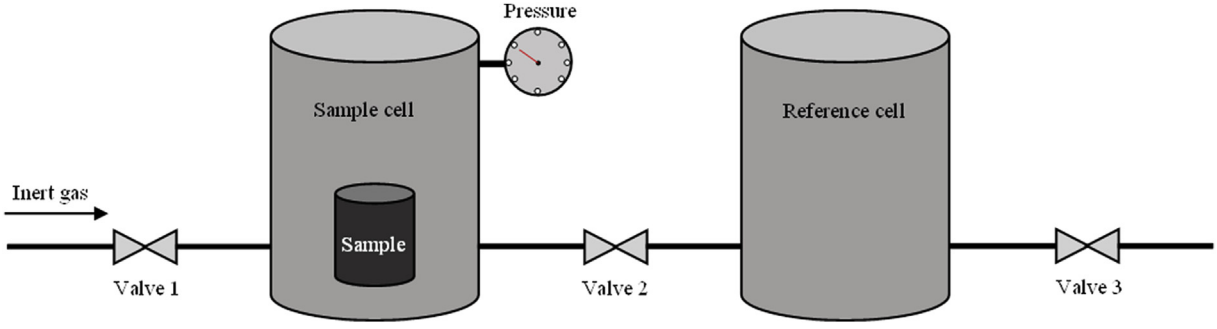

Fig. 11. Schematic of a typical gas displacement Pycnometer.

[67] used a 1 liter steel container for pellets of less than 12 mm in diameter. Jackson et al. [13] and Mani et al. [17] used a funnel above the center of the container in order to have a more homogenous filling.

2.4.4. Summary

Particle density is an important factor in densified material formulation and quality while the bulk density is of high importance in the packaging, transport, and marketing [31]. Mainly, the bulk density depends on the particle density and the voids ratios. Nonetheless, bulk density is not an inherent property of the material and should be considered as a dynamic property rather than a static one. Regardless of the material components and densification process, bulk density depends on the test procedure and examiner accuracy [71]. Pellets or briquettes rearrangement in the container during the test, PSD, container material and surface smoothness, and container filling method are the factors affecting the bulk density. Although using different methods rather than standards may increase the accuracy, it creates difficulties in comparing the results of different methods. Consequently, the bulk material values in literature are not comparable unless a unique method is used.

2.5. Hydrophobicity

Most of the material being densified might adsorb moisture from the environment after densification. Biomass, due to its inherent hydrophilic nature [72] is a well-known example. Increasing the biomass moisture content might degrade the material and will decrease the energy content on an as received basis [72,73]. Several research works have been carried out to increase the biomass hydrophobicity by means of torrefaction, steam explosion, and by increasing densification temperature [4,5,64]. Some methods had partially increased the hydrophobicity, however, still the material adsorbs moisture when exposed to a high humid environment (60–80%) [60].

Generally, the hydrophobicity tests could be divided into two different methods; firstly to position the sample in a humid environment (moisture adsorption); secondly to immerse the sample in water (water resistance). The former simulates the storage and transportation condition under humid weather conditions while the latter simulates rain exposure conditions. Many research have been done in order to investigate the hydrophobicity of different bio-material under different conditions of which some are discussed in the following.

2.5.1. Moisture adsorption

The moisture adsorption test methods in literature could be divided into two major groups: inside (laboratory) and outside. Mostly, humidity chambers are used in laboratory tests to determine the amount of moisture that a sample can adsorb under constant temperature and humidity conditions. Peng et al. [74], Jiang et al. [3], and Li et al. [15] used a humid chamber at 30 °C temperature and 90% relative humidity to determine the moisture adsorption of biomass and torrefied pellets. Pellets were pre-dried at 105 °C for 24 h. During the experiments, the sample mass was measured at sequential time intervals until reaching a constant level. The increase in the pellet mass was reported as the amount of moisture adsorption. For the saturated moisture measurements, Peng et al. [74] placed the pellets in the chamber at the temperature of 20–35 °C and relative humidity from 40 to 95%. Hu et al. [4], used the above-mentioned procedure while setting the humidity at 70% for pyrolysed woody biomass. Rhén et al. [41] also followed the same procedure for Norway spruce pellets, however, before starting the moisture adsorption experiments they let the pellets equilibrate in a 30% humidity environment at 23 °C.

Kambo and Dutta [5] used a relative humidity of 48–52% and a temperature of 22–23 °C for 24 h to determine the equilibrium moisture content (EMC) of raw and pre-treated miscanthus pellets. After the test, samples were dried at 103 °C for 16 h. The mass difference before and after drying was expressed as the EMC.

As an example of the outside experiments, Bergström et al. [14] placed batches of pine sawdust pellets (five randomly selected pellets in each batch) outside covered with a roof for 14 days. The environmental conditions were recorded and showed the temperature between −20 and 0 °C and humidity of 74–100%. Then the moisture adsorption was reported based on the mass difference before and after the test. The results showed around 10% moisture adsorption for all the samples.

Comparing the laboratory environment chambers and outdoor storage, the primary advantage of the chamber is that the temperature and humidity could be set at a constant level and could be adjusted according to the atmosphere of the location. However, the fluctuations in the real atmosphere are neglected while in the outdoor storage the material meets the real weather conditions. Anyway, using the chamber is more common because the experimental conditions are more controllable.

2.5.2. Water resistance

The ability of a material to resist water is normally measured by the water immersion test. Similar to the moisture adsorption tests, water resistance measurements in literature can be divided into two major groups: inside (laboratory) and outside conditions. Water immersion is used when the material does not degrade in water, otherwise, the test is disrupted. Richards [31], set an inside water immersion test which immerses a single briquette in a cool water bath for up to 30 min. The physical consistency of the materials was checked at 10 min intervals by finger pressure. Intact materials were weighted and the proportion of adsorbed water was determined. Then he defined the water resistance index (WRI) as an indication of moisture adsorption and proposed a minimum value of 95 for WRI would be acceptable for most briquettes.

$$WRI = 100 - (\%\ water\ absorbed\ after\ 30\ min\ immersion) \quad (17)$$

Bazargan et al. [22] followed the procedure above for palm kernel shell bio-chars. Sengar et al. [23] measured the water adsorption of biomass briquettes by immersing them in water height of 25 mm at 27 °C for 30 s and measured the resistance to water penetration using the equation (17).

Kambo and Dutta [5] following the method described in Pimchuai et al. [75], immersed torrefied and hydrothermally carbonized pellets in water for 2 h. Then the pellets were removed from the water and excess water removed by using an adsorbent paper. After that, the pellets were put in a controlled environment of 48–52% relative humidity and 22–23 °C temperature for 4 h. The moisture adsorption was determined by the change in the pellet mass.

2.5.3. Summary

Biomass-based materials are hydrophilic in nature, however, different techniques may improve their hydrophobicity. In some cases, even a small amount of moisture may notably decrease the quality. So far, there is no standard method to accurately measure the moisture adsorption in different well-defined conditions and there is a need for that. Developing a standard method could simultaneously eliminate the concerns about the suitability of the storage place and reduce extra costs due to overestimation of the environmental conditions.

3. Factors affecting the physical properties of densified material

Abdollahi et al. [36], in their review study , have determined the possible factors to manufacture animal feed pellets with high material quality. These factors include diet formulation, binder addition, manipulation of PSD, manipulation of steam in densification process, press setting, decrease of production rate, and manipulating cooling and drying. Stelte et al. [35], in their review paper, pointed out the process variables affecting the densification process and products, namely: the moisture content, temperature, particle size, press channel dimensions, and pelletizing pressure. However, this paper points out the recent findings in the area of densification and systematically addresses the effect of different variables from an integrated perspective.

Regardless of the material type, generally the affecting factors on the physical properties of densified products can be classified into four major groups namely the raw material, preparation conditions, densification process, and storage conditions. The effects of these factors are discussed in details in the following sections.

3.1. Raw material

3.1.1. Raw material composition and blends

The raw material components play a key role in determining the physical properties of densified material. The presence of lignin, proteins, and starch help to increase the inter-particle bonding, increasing hardness and durability [10].

Several studies showed that blending biomass of different raw materials could improve durability and compressive strength of densified products [24,28]. Mitchual et al. [43] reported that mixing 10% corn cobs with biomass sawdust of C. Pentandra at compressive pressures of 30–50 MPa significantly improved the compressive strength of briquettes compared to briquettes made of only single biomass type (see Table 2). The compressive strength of pure corn cobs and pure C. Pentandra was 0.54 and 44.58 N mm^{-1}, respectively, while the compressive strength of the mixture was 59.22 N mm^{-1}.

3.1.2. Moisture content

Moisture content is a key process factor in the production of densified material. Bazargan et al. [22] reported that it is not possible to make strong pellets without the presence of moisture. Abdollahi et al. [18] have studied the effect of moisture on the quality of animal feed pellets made from wheat. They concluded that at the densification temperature of 60 °C the addition of 24 g kg^{-1} moisture (i.e. 2.4 wt.%) increases both hardness and pellet durability index (see Tables 2 and 4).

Many studies have investigated the effect of moisture content on the durability of densified biomass [6,13,19,22,51,60,69]. They unanimously argue that by increasing the moisture, material strength first increases and then decreases. The optimum value depends on the biomass species. For example, for peanut hull pellets it was reported that the presence of 9.1% moisture produced the most durable pellets with the durability value of 90.3% [60]. Jackson et al. [13] made pellets with four different biomass spices: miscanthus, switch grass, corn stover, and wheat straw and reported that the least necessary moisture value for pellet making is 20% while the most durable pellets (durability of 90%) contain 25% moisture. In the study of palm kernel shell biochar, Bazargan et al. [22] obtained the optimum moisture of 30% for the highest tensile strength of 0.035 MPa for briquettes made at 60 MPa pressure. They also found that adding 20% water along with 10% starch increased the tensile strength of palm kernel biochar more than 100 fold. Umar et al. [19] studied the physical properties of extruded aquafeed and observed the maximum durability of 86.6% at a moisture level of 40%.

Tabil et al. [51] studied the effect of moisture content on the Meyer hardness of two different alfalfa cubes having 7.1, 10.8, and 14.3% moisture and reported that regardless of the cube type and probe size, the Meyer hardness decreased by increasing the moisture.

Zainuddin et al. [6] made pellets of pineapple to use as the animal feed. They made the pellets using four moisture levels of 35, 40, 45, and 50% and observed a minor difference in both bulk density and friability of pellets. However, other researchers [17,76–78] reported a negative influence on bulk density by increasing the moisture content.

In addition to the above-mentioned literature results, Huang et al. [79] concluded that although the presence of moisture is vital for the densification process, the optimum amount varies depending on the material type. Moreover, they believed that the effect of moisture content might depend on other factors such as temperature and pressure, therefore, that should not be investigated alone. More research on the effect of simultaneous factors is necessary to fully understand their influence on the final product quality.

3.1.3. Particle size distribution

Bazargan et al. [22] densified bio-chars of different particle size using 0.7, 3, and 7 mm sieve sizes. They concluded that the finer particles lead to more smooth surface and higher tensile strength compared to coarse particles (see Table 2). Muazu and Stegemann [21] used a PSD of less than 2 mm in their study and reported that the lower PSD leads to less relaxation. Lindley and Vossoughi [26] in their study used particles of less than 2 mm (from 0.004 to 2 mm) and concluded that smaller particle sizes make stronger briquettes. Mani et al. [17] used PSD of between 0.075 and 3.2 mm for wheat straw, barley straw, corn stover, and switchgrass and reported a slight increase in density for all pellets by decreasing the particle sizes, except for the wheat straw.

Gilbert et al. [20] observed the more compressive strength of pellets made by cut switchgrass (10–70 mm length) in comparing with shredded (< 4 mm length) and torrefied switchgrass. They believed that the reason lies in the interlocking of long strands of cut switchgrass which act as an additional binding alongside the lignin effect. Mitchual et al. [29] also observed more strength for the samples made by using bigger particle sizes. They declared that their results contradict the other researchers' results. They believed that as the smaller particles show more surface area, at higher temperatures starch gelatinization occurs, making the pellet stronger. However, because they performed the experiments at room temperature the effect of starch gelatinization disappeared.

As a conclusion, the effect of particle size depends on the mechanical interlocking of particles. Larger particle sizes cause increased interlocking, creating a stronger bonding. At higher temperatures, the greater surface area provided by finer particles increases the bonding opportunities as well by activating different bonding phenomena such as starch gelatinization, lignin glass transition, and protein denaturation.

3.2. Pretreatment conditions

Biomass pretreatment is carried out to improve the physical and/or chemical properties of the material. Pyrolysis, torrefaction, and hydrothermal carbonization are some examples of the common pretreatment techniques for improvement of biofuels properties [16,20,59]. Due to the physicochemical changes, the material behavior after densification will also change. For example, torrefaction is reported to change the properties of biomass from hydrophilic to hydrophobic [80]. Here, it should be noted that the pretreatment is not always taken into consideration as an affecting factor on densified material properties because in some cases the raw material is densified without any pretreatment.

Liu et al. [46] studied the effect of hydrothermal carbonization of woody and agro-residue biomass on the physical properties of pellets and found that the compressive strength of all the samples increases notably after hydrothermal carbonization (see Table 2). The moisture uptake of all the materials also decreased by carbonization, an indication of changing the hydrophilic structure to hydrophobic.

Kambo and Dutta [5] made pellets from raw, torrefied, and hydrothermally carbonized miscanthus (at three different temperatures of carbonization) and found that the compressive strength of the pellets decreased by increasing the carbonization temperature while pellets made from torrefied biomass show the least compressive strength compared to hydrothermally carbonized and raw biomass (see Table 2). Wu et al. [81] also observed the same results and reported that hydrothermally treated cotton stalk and wood sawdust show higher compressive strength than torrefied cotton stalk and wood sawdust. Kambo and Dutta [5] suggested that the lower compressive strength of torrefied biomass is due to the observed pores inside the material structure. Moreover, as stated by Liu et al. [46] hydrothermal carbonization increases the hydrophobicity, resistance against water immersion and increases the grindability of pellets. Peng et al. [74] and Li et al. [15] also reported similar results as Kambo and Dutta [5] where

they found torrefied material more difficult to compress into dense and strong pellets compared to non-torrefied pellets.

Hu et al. [4] made various pellets by using pyrolysed biomass at the temperatures of 250 °C, 350 °C, 450 °C, 550 °C, and 650 °C at 128 MPa densification pressure and 35% moisture content. Considering the bulk density, first, they observed a slight decrease and then a notable increase by increasing the pyrolysis temperature. The trend for the compressive strength was similar to the bulk density. For biomass pyrolysed at 250 °C, the compressive strength was around 5 MPa and decreased to around 4 MPa for pyrolysed biomass at 350 °C and then sharply increased to around 15 MPa for 650 °C pyrolysis biomass temperature. They also concluded that the effect of pyrolysis temperature on the pellet properties was dominant over the moisture content.

3.3. Densification process

The effecting parameters on densification can be distinguished into the densification process temperature, pressure, dwell (holding) time, press shape and length, and cooling and drying. Below, a detailed explanation of the effects of each of these parameters is given.

3.3.1. Press temperature

The press temperature is reported to have a high influence on the product density and hardness [8,74]. The relation between die temperature and pellet hardness and density lies in the raw material components. As an example, lignin is one of the main natural binders found in the biomass species. Increasing the material temperature helps lignin to reach the glass transition temperature (around 100–140 °C) thus improves the bonding mechanism and hardness [74]. The die temperature is normally elevated on purpose, however, it usually abruptly increases during compression due to particle-wall frictions [15].

Li et al. [15] observed an around 4.7 fold increase in hardness of untreated sawdust by increasing the die temperature from 70 to 170 °C and the Meyer hardness increased from 1.44 to 6.81 N mm^{-2}.

Some researchers believed that a compression temperature higher than room temperature is crucial for making pellets with high durability [20,26]. Carone et al. [8] believed that in order to generate highly durable olive residue pellets with the highest density, a minimum temperature of 150 °C is required. Lam et al. [50] reported that for the Douglas fir species the optimum die temperature for the hardest material is 200 °C. Peng et al. [74] stated that to obtain the same hardness as raw biomass pellets, a die temperature of at least 230 °C is required for the torrefied pellets. Verhoeff et al. [82] also reported that torrefied pellets compressed at a die temperature of 225 °C result in a durability of about two times greater than the raw biomass pellets densified at 100 °C.

Considering these reports, one may conclude that the compression temperature to make the most durable pellets should exceed 150 °C, however, high temperature might affect the raw material structure to increase the brittleness and lead to the loss of a big portion of moisture content [20]. Moreover, one should pay a serious attention to the fact that in most of the aforementioned studies a single or laboratory scale densifier rather than a pilot or an industrial scale piece of equipment was used. Segerström and Larsson [83] showed that although a single pelletizer could mimic a pilot scale densification process, the effect of die temperature on pellet density is inconsistent for single and pilot scale. In single pelletization setup, the temperature has a positive effect on the pellet density, however, in pilot scale process the pellet density has a negative correlation with the die temperature.

3.3.2. Pressure and residence time

Several studies investigated the effect of compression pressure on density and hardness of pellets and briquettes [1,4,17,20–22,25–27,29,41,43,65,74,84]. Fig. 12 shows the applied pressure intervals. According to these literature sources, density increases by increasing the compression pressure, however, in some cases it was insignificant. The effect on the hardness (compressive strength or tensile strength) was complicated. At compression pressures of 1 to around 50 MPa, hardness increases by increasing the pressure. For example, Chin and Siddiqui [25] reported that the shear strength increased around 3.5–7.3 fold by increasing the press pressure from 1 to 10 MPa for biomass briquettes of different origins. Nonetheless, there exist some exceptions, for example, Al-Widyan et al. [27] reported an optimum pressure of 35 MPa in their studies for adequate durability of olive cake briquettes. For higher pressures of up to 130 MPa usually there is a maximum at which the material hardness shows the highest value. For instance, the reported optimum pressure for gasified palm kernel shell was 60 MPa [22], and for wood biochar 128 MPa [4]. Peng et al. [47] and Demirbas and Sahin-Demirbas [65] examined higher pressures of 125–249 MPa and 300–800 MPa, respectively, and concluded that the effect of pressure on the material hardness is very low, i.e. the hardness is less sensitive to the pressure.

Li and Liu [1] investigated the effect of residence time (also known as holding, dwell, and retention time) from 0 to 60 s and observed a 5% increase in density when increasing the holding time from 0 to 10 s. The density increased as the time increased up to 20 s and after that no significant increase in density was observed. However, they observed no effect of holding time at a high pressure of 138 MPa. Chin and Siddiqui [25] also reported dwell time between 20 and 60 s as the optimum for different biomass spices of sawdust, rice husk, peanut shell, coconut fiber, and palm fiber. For olive cake briquettes, Al-Widyan et al. [27] reported that neither durability nor density increases by applying dwell time of between 5 and 20 s, therefore dwell time should not exceed 5 s. Bazargan et al. [22] concluded that at high pressure densification the holding time has almost no effect on the material properties.

3.3.3. Binder

Generally, binders help stronger bonding between particles, thus increasing the hardness and durability of densified products [26,59]. The presence of structurally incorporated binders, such as lignin and proteins improves the hardness and durability of the densified material, especially at high levels of pressure and temperature [3,30,85], however, a high lignin content is reported to be responsible for the brittle structure of densified material [5]. In many cases, the structurally incorporated binders are not enough to make a highly durable material, thus the addition of an external binder is vital. Addition of 10% starch and 20% water as binder reported to increase the biochar pellet hardness more than 100 times [22].

The addition of binder could be as easy and cheap as adding water [86] or it could be a kind of biomass, starch, protein, glycerin, etc. Mostly, the addition of binder increases the total cost of the process and in some cases it may affect negatively the combustion behavior and density of densified fuels [5]. Muazu and Stegemann [21], in their study of preparing the rice husks and corn cobs briquettes used starch as binder and found that starch inflicted the particle swelling which notably decreased the relaxed density.

Choosing the appropriate binder type and dose is of vital importance for densified material preparation. Not surprisingly, Järvinen and Agar [48] observed lower quality when using wheat flour as a binder to prepare pellets from torrefied pine. In general, they observed lower density, durability, hardness and energy density and more moisture uptake when adding wheat flour as a binder. For some animal feed pellets, Abdollahi et al. [18] also observed a slight decrease in compressive strength by the addition of a binder along with moisture compared to adding moisture alone (see Table 2).

3.3.4. Press shape and press channel length

According to Richards [31], the shape of a densified material affects the durability i.e. regardless of the other factors, materials with sharp edges show lower abrasion resistance than those with a round shaped

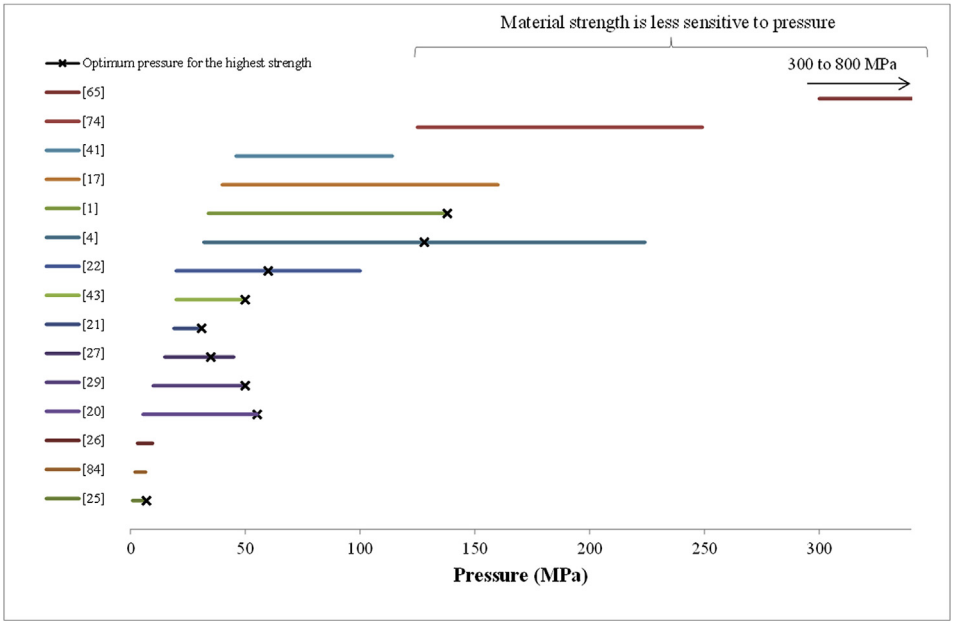

Fig. 12. Densification pressure domain reported in literature.

edge.

Thrän et al. [73] believed that pellets with a 6 mm diameter comprise the highest durability, while pellets with a diameter of 8 mm increase the production capacity. They also reported that for torrefied softwood and torrefied herbaceous biomass die aspect ratio (the ratio of diameter to length) is an affecting factor on durability. Heffner and Pfost [87] showed that durability increases as the aspect ratio increases.

In the other study, the effect of press channel length on pelletization of torrefied scot pine was investigated [88]. Three channel lengths of 25, 30, and 35 mm representing the aspect ratio of 3.125, 3.75, and 4.375, respectively, were used. They reported that a 35 mm channel length yielded high and fluctuated the mill motor current, thus they eliminated this channel length from the investigations. They also reported that it was not possible to pelletize the biomass torrefied at 291 °C with a channel length of 25 mm. The reason is not clearly stated in their work. However, for samples torrefied at higher temperature of 308 and 315 °C the pellets were successfully made using the same channel length. Comparing the effect of channel length on the bulk density, they reported around 20 kg m^{-3} increase in bulk density with increase of channel length for 1 mm.

3.3.5. Cooling and drying

Normally, densified material leave the densification process at temperatures ranging from 60 to 95 °C and moisture content between 12 and 17.5% on a wet basis while the desired temperature and moisture is normally 5–8 °C higher than the ambient temperature and 5–8%, respectively [36,89]. If the material is not cooled and dried properly, it may lose its quality and result in heating, combustion, and caking in the post transportation and storage. Cooling time is an important factor in determining the material quality. According to Maier and Bakker-Arkema [89], the cooling time may take 4–15 min, however, it should be noted that choosing the optimum cooling time is of high importance in terms of material quality. Too quick cooling may result in cooling the outer layer while the inner layer remains warmer resulting in stresses in the material followed by the crack formation in the outer layer and a decrease of the mechanical strength. On the other hand, a too long cooling period may result in a too dry material which increases the brittleness and reduced quality [36].

3.4. Storage conditions

Storage is one of the most important parts of a supply chain [72]. The storage time and atmospheric conditions (temperature and humidity) are of crucial importance for any kind of densified bio-material.

Many authors investigated the effect of atmospheric conditions on the physical properties of densified material [3–5,14,15,41,47]. All researchers argued that storing in humid condition increased the moisture content of biomass-based material. Weatherstone et al. [64] stored torrefied spruce and poplar pellets outside in the stockpiles of 1–4 tonnes for more than 6 months and observed large moisture adsorption and degradation of the upper layer (around 10 cm). They concluded that outside uncovered storage for a long period of time will deteriorate the torrefied biomass quality.

The volume of the material might expand during the storage time. Jiang et al. [3] have made pellets of Chinese fir, camphor, and rice straw and observed 0.31–1.34% volume expansion (with or without adding binder) after two weeks of the storage. The interesting point was that they reported around 1.25% fine particles separated from the pure biomass pellet surface during the storage time. Elastic recovery of the biomass particles and weak bonding were believed to cause this phenomena.

4. Discussion

As shown in this review, the reported quality characterization methods mostly do not follow a standard procedure. As a result, the quality values reported are hardly comparable to other literature sources making the assessment difficult or impossible. Furthermore, the use of dissimilar units make it even harder to compare the results. For example, as presented in Table 2 the compressive strength values are reported in already different units of N, N.mm^{-1}, and MPa.

Researchers showed that even some of the existing standards are not capable of testing the quality parameters for a range of material characteristics. For example, Weatherstone et al. [64] reported that the EN15210-1 [62] standard for the durability measurement of untreated wood pellets is not appropriate for materials with high moisture content and requires further modifications. In addition, durability testers measure fines generation when abrasive forces are encountered, however, the scale of the compressive forces applied does not match with

the large-scale transportation conditions. Therefore, the existing standards require development based on the real conditions. The future investigations of the measurement methods of densified material quality parameters should focus on development, preparation, and use of dedicated standards in order to unify the test procedures and make different results comparable.

Dust generation is a crucial factor in determining a densified material quality, especially from a health, safety, and the environment (HSE) perspective. Dust is detrimental from three points of view; material loss, equipment fouling issues, and environmental problems. Dust generation capability during large-scale transportation can be investigated by several laboratory scale experiments, however, this is very difficult and requires dedicated facilities pushing the need for standards.

Regarding the existing literature, many researchers used the One-Variable-At-a-Time (OVAT) approach to investigate the factors affecting the quality parameters of densified bio-materials [4,6,13,20,25,28]. In this method, the effect of each factor is individually investigated while the other factors are kept constant. Not only using OVAT increases the number of experiments and requires resources, but also it is often not reliable and may lead to incorrect results. According to Jiju [90], major problems in industrial process optimizations are mostly due to the interactions between the factors rather than the effect of individual factors. Changing only one variable at different levels might be advantageous to reach the optimum value of a specific parameter or when the other factors are less important in process optimization. However, as we showed here, there are many factors involved in bio-material densification which play key roles in the product quality. The future research in this field may focus on discovering the most affecting interactions and optimization of different parameters, simultaneously. Nevertheless, one should pay attention to the fact that the conclusions presented in chapter 3 are based on the assumption that all the quality parameters are assessed in the same way, i.e. using the same methods and devices. However, as shown in chapter 2, the quality assessment method has an extreme impact on the results of compressive strength, durability, and density.

5. Conclusions and outlook

In order to measure the quality parameters of densified bio-materials, numerous devices and customized methods have been used by researchers. We showed that results reported in literature are not comparable unless the same devices, processing conditions and methods are applied. Although all the quality parameters shown here are of high significance in transportation, handling, and storage of densified bio-material, there exist only a few standard methods for; durability and density. Therefore, there is an urgent need for developing new standard methods for compressive strength determination (including hardness and bending test), impact testing, and characterization of hydrophobicity.

Considering the existing quality standards, there is no clear relationship between the experimental results and issues of bio-solids handling under real conditions. The existing standards can classify different pellets based on their fines generations in a laboratory condition, however, they provide no information about the particle breakage or the amount of fines created during the whole supply chain. A suitable standard method should mimic the real transportation and storage issues by considering the impact, compressive and abrasive forces on the materials simultaneously.

Besides experiments to assess the physical properties of densified bio-materials, computer modeling tools such as discrete element method (DEM) can be applied to decrease the experimental cost and time. In DEM, individual particles can be modelled to represent the material behavior of a bulk solid. For example, Schott et al. [37] and Mahajan et al. [91] compared the durability of wood pellets in different conditions using DEM and found reliable results. Use of these numerical methods could accelerate the design and optimization of transportation and storage facilities. Anyway, using DEM in densified bio-material is in its initial stages of research and requires more studies.

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